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Lockheed Engineering and Management Services
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DIRECT/DELAYED RESPONSE PROJECT: FIELD OPERATIONS AND QUALITY ASSURANCE REPORT FOR SOIL SAMPLING AND PREPARATION IN THE NORTHEASTERN UNITED STATES

VOLUME II PREPARATION

by

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The Direct/Delayed Response Project Soil Survey includes the mapping, characterization, sampling, preparation, and analysis of soils in order to assess watershed response to acidic deposition within various regions of the United States. Soil samples collected by sampling crews in the Northeastern region were processed at preparation laboratories before being sent for analysis at analytical laboratories. Volumes I and II summarize the procedural and operational compliance with protocols used by the sampling crews and by the preparation laboratories, respectively. Deviations from protocols and difficulties encountered are identified and discussed. Recommendations are made for program improvement.

In general, soil sampling activities during the survey proceeded as planned. A review of the soil data suggests that the integrity of the soil samples was maintained during the preparation activities. In most cases, sampling crews and laboratory personnel adhered to protocols.

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ABSTRACT

The Direct/Delayed Response Project Soil Survey includes the mapping, characterization, sampling, preparation, and analysis of soils in order to characterize watershed response to acidic deposition within the Northeastern region of the United States. Soil samples collected by sampling crews were delivered to preparation laboratories where the samples were processed and were organized into batches for shipment to analytical laboratories. This document summarizes the procedures and assesses the compliance with protocols used at the preparation laboratories. Difficulties at the laboratories are discussed, and recommendations are made for program improvement.

In general, the preparation laboratories observed protocol. Soil sample integrity appears to have been maintained at the preparation laboratory.

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SECTION 1

INTRODUCTION

SUMMARY

The U.S. Environmental Protection Agency (EPA), in conjunction with the National Acid Precipitation Assessment Program (NAPAP), has designed and implemented a research program to predict the long-term response of watersheds and surface waters in the United States to acidic deposition. Based on this research, each watershed system studied will be classified according to the time scale in which it will reach an acidic steady state, assuming current levels of acidic deposition. The Direct/Delayed Response Project (DDRP) was designed as the soil study complement to the aquatic resources program.

As part of the DDRP, four preparation laboratories were established in the Northeastern region of the United States to process soil samples and to perform preliminary analyses on these samples. The preparation laboratories were located within the soil science departments at the following land grant universities:

- University of Massachusetts, Amherst, Massachusetts
- University of Connecticut, Storrs, Connecticut
- University of Maine, Orono, Maine
- Cornell University, Ithaca, New York

Each laboratory was under the direction of a laboratory manager, who was a soil scientist and a member of the university faculty. The manager was responsible for ensuring that the integrity of the soil samples was maintained after the samples arrived at the preparation laboratory. Laboratory personnel were required to comply with protocols specified for DDRP (Appendix A), which are referred to as the protocols throughout this report. Each laboratory manager employed at least one full-time assistant as well as a number of college students. Most of the student assistants were majoring in soil science, with the remainder studying related fields.

The preparation laboratories performed the following analyses on the bulk samples collected by the sampling crews:

- percent moisture (air dry)
- percent rock fragments (in the 2- to 20-mm fraction)

- qualitative test for inorganic carbon

- clod analysis for bulk density

Laboratory personnel prepared one-kilogram analytical samples derived from homogenized air-dry bulk samples. The analytical samples were labeled and placed into batches. The samples then were randomized within each batch. In addition to the routine samples, each sampling crew collected one duplicate soil sample per day for quality assurance (QA) purposes. Natural audit samples and a preparation laboratory duplicate also were placed into each batch for QA purposes. The batches then were shipped from the preparation laboratories to various analytical laboratories contracted by the EPA.

The results of analyses performed by the preparation laboratories were recorded on a Form 101 for each batch of samples. Copies of the completed Form 101 were required to be sent to the data base management personnel at Oak Ridge National Laboratory (ORNL), to the QA staff at the EPA Environmental Monitoring Systems Laboratory in Las Vegas (EMSL-LV), and to the EPA Environmental Research Laboratory in Corvallis (ERL-C) within 24 hours of batch shipment to the analytical laboratories.

Because the bulk density analysis was not completed when batches were sent to the analytical laboratories, copies of the Form 101 were not always sent within the specified time limit. Copies specifically were not sent to the analytical laboratories because the forms identified the types of soil horizons being analyzed. Instead, analytical laboratories received copies of the shipping form (Form 102), on which the sample codes from the Form 101 had been removed. Each sample on the Form 102 was identified only by batch and sample numbers in order to disguise the identity of the soil horizon from which the sample originated.

Any surplus soil material at the preparation laboratories was archived and placed in cold storage. After the sample preparation and shipping activities were completed, the archived samples were shipped to Las Vegas via refrigerated truck and were placed in long-term cold storage facilities.

Information concerning sample collection, labeling, and analysis was documented in sample receipt and sample processing log books. These log books later were sent to EMSL-LV for reference during the data verification procedure.

Quality assurance/quality control (QA/QC) measures were applied in order to maintain consistency in the soil preparation protocols and to ensure that soil sample analyses would yield results of known quality. Personnel at the preparation laboratories received training on the analytical methods and soil preparation procedures. QA staff from EMSL-LV conducted on-site systems audits of the preparation laboratories. Weekly communication between QA personnel and laboratory personnel was established to identify, discuss, and resolve issues. Representatives of three of the preparation laboratories attended an exit meeting held in Las Vegas on January 6 and 7, 1986. The purposes of the meeting were to review the mapping, sampling, and preparation activities, resolve any remaining issues, and generate suggestions for future surveys.

OBJECTIVES

This document reports the results of the preparation laboratory operations and QA program for the Northeastern DDRP Soil Survey. Difficulties encountered with the data or methods are identified, and suggestions for corrective actions are made. The preparation laboratories are identified by number rather than by name in order to prevent disclosure of their identity. Information concerning the specified protocols for soil preparation can be found in the protocols (Appendix A).

SECTION 2

SAMPLE PREPARATION METHODS AND ANALYSIS

The detailed methods and analytical procedures used in soil preparation activities for the Northeastern soil survey are given in the protocols (Appendix A). Brief explanations of the methods and procedures for the preparation laboratory analyses are given below, including two corrections to the protocols. The methods are discussed in sequential order as performed in the preparation laboratories.

SAMPLE PROCESSING AND ROCK FRAGMENT DETERMINATION

Each bulk sample is spread on a tray to air dry until constant weight is achieved. This is confirmed by comparing the weight of a subsample on two consecutive days. If the weight change is less than 2.5 percent, the sample is considered air dry.

After recording the weight of the air-dry bulk sample, the soil is passed through a No. 10 mesh sieve to collect the less than 2-mm soil fraction. The rock fragments that are retained on the sieve constitute the 2- to 20-mm pebble fraction. The fragments are weighed, are bagged, and are placed in storage. The fragment weight is divided by the total air-dry sample weight and is multiplied by 100 to obtain the percent rock fragments.

A Jones-type 3/8-inch riffle splitter is used to homogenize the less than 2-mm fraction of the sample. The soil is placed through the riffle splitter at least seven times in succession. One-half of the sample is placed into a plastic bag for archiving, and the other half is placed through the riffle splitter until an approximately one-kilogram sample is obtained.

QUALITATIVE TEST FOR INORGANIC CARBON

One gram of soil is placed in the well of a porcelain spot plate, is saturated with deionized water, and is stirred to release any entrapped air. Three drops of 4N HCl are added to the sample while the analyst uses a stereoscope to observe the chemical reaction.

BULK DENSITY DETERMINATION

Replicate soil clods are collected by the sampling crews. Upon arrival at the laboratory, the clods are weighed and are dipped in a 1:4 or 1:7 saran: acetone mixture. The clods are suspended from a line, are allowed to dry, and are reweighed. This procedure is repeated, usually three or four times, until each clod is impervious to water.

Approximately 800-mL deionized water contained in a one-liter beaker is de-gassed by boiling, is allowed to cool to room temperature, and is tared on a balance. Each clod is submerged in the water to determine its weight displacement on the balance. The clods are placed in a drying oven for 48 hours and, after cooling, are weighed. A two-hour heat treatment in a 400°C muffle furnace vaporizes the saran, then the clods are cooled and are reweighed. Each clod is crushed and is passed through a No. 10, 2-mm mesh sieve. The rock fragments are weighed, and the data is used to correct for the rock fragment content.

Bulk density is defined as the mass of a unit volume of soil particles and pore space expressed as grams per cubic centimeter (g/cm^3). Bulk density in mineral soils normally ranges from 1.0 to 1.8 g/cm^3 (USDA-SCS, 1983).

Two of the bulk density algorithms were given incorrectly in the protocols. The first algorithm originally was written as:

$$BD_{FM} = \frac{M_{OD} - [M_{CF} + M_{TS}\,(0.85)]}{\dfrac{M_V}{rH_2O_T} - \dfrac{M_{CF}}{2.65} + \dfrac{M_{TS}}{1.30}}$$

where:

BD_{FM} = bulk density in g/cm^3
M_{OD} = oven-dry clod weight
M_{CF} = rock fragment weight
M_{TS} = saran coating weight
0.85 = air-dry to over-dry saran weight conversion
M_V = clod weight in water
rH_2O_T = density of water at laboratory temperature, T
2.65 = density of rock fragments
1.30 = density of saran

Using this algorithm, the saran volume would be added to the clod volume rather than subtracted, thereby underestimating the bulk density value.

The correct algorithm is as follows:

$$BD_{FM} = \frac{M_{OD} - [M_{CF} + M_{TS}\,(0.85)]}{\dfrac{M_V}{rH_2O_T} - \left(\dfrac{M_{CF}}{2.65} + \dfrac{M_{TS}}{1.30}\right)}$$

The second algorithm was used to estimate the air-dry saran weight and was incorrectly written as:

$$M_{TS} = \frac{X\ (M_a - M_1)}{a - 1}$$

where:

M_{TS} = air-dry saran weight
X = total number of coatings (field and laboratory)
M_a = clod weight after final coating
M_1 = initial clod weight after unpacking
a = number of laboratory coatings

The correct algorithm is as follows:

$$M_{TS} = \frac{X\ (M_a - M_1)}{a}$$

SECTION 3

PREPARATION LABORATORY OPERATIONS

Detailed discussions of the operations performed by the preparation laboratories follow. Deviations from the specified protocols and difficulties with the methods are noted.

SAMPLE STORAGE

The specified temperature for storage of soil samples was 4°C, which was monitored on a daily basis. All preparation laboratories had adequate facilities to store the samples at the required temperature.

An ammonia leak was discovered in the cold storage facility at Laboratory 3. The Nessler Reagent Colorimetric test (Greweling and Peech, 1960), a test for ammonium content, was used to assess the possible contamination of soil samples that were exposed (unbagged), single-bagged in plastic, and double-bagged in plastic (Appendix B). The conclusions of the experiment were that the leak had no effect on single- or double-bagged samples. All samples processed by Laboratory 3 were double-bagged in cold storage.

EQUIPMENT

EMSL-LV shipped sampling equipment to the preparation laboratories where laboratory personnel were responsible for distributing equipment to the sampling crews. The crews usually picked up supplies while samples were being delivered to the cold storage facility. All supplies taken by the sampling crews were required to be listed in the equipment log book.

Laboratory personnel were tasked with mixing the saran solution used for coating clods in the field. Equipment shortages were reported to EMSL-LV during the weekly conference calls. After the soil preparation activities were completed, leftover supplies were inventoried and were shipped to EMSL-LV for storage. A list of the equipment provided to the sampling crews can be found in Appendix A.

RECORD KEEPING

Preparation laboratory data for the Northeastern soil survey were placed in log books or on forms produced by individual laboratories. Each preparation laboratory maintained the following log books:

- Label A - Labels that the sampling crews had placed on the inner soil sampling bags were removed from the bags and were affixed to pages of the Label A log book.
- Clod Labels - Labels that the sampling crews had attached either to the clods or to the clod box were removed and were affixed to pages of the clod label log book.
- Sample Receipt - Information gathered upon receipt of the soil samples from the field.
- Percent Moisture - Raw data from the moisture analyses.
- Rock Fragment - Raw data from the rock fragment analyses.
- Bulk Density - Raw data from the bulk density analyses.
- Inorganic Carbon - Raw data from the inorganic carbon analyses.
- Sample Processing - Tracking of the soil samples through the various preparation activities.

Because a standard format for record keeping was not specified, there was great variation from laboratory to laboratory. As a result, verification of data was a difficult process.

SAMPLE DRYING

There were no deviations from the specified sample drying procedure. Because the procedure was not sufficiently detailed, the sample drying operations did not proceed as efficiently as possible. Laboratory 1 identified wet organic soils of which the outer layer of the bulk sample had dried to a hard crust, thereby inhibiting the flow of air to the rest of the sample. Laboratory 3 used cafeteria trays on which to dry the samples and this resulted in the collection of water on the tray surface beneath the sample. Both situations created conditions favoring microbial growth that may have altered the composition of the affected soil samples. Specifications for the drying area did not stipulate any ventilation requirements to accelerate the drying process. In addition, there were no requirements to limit access to the drying room or to prevent soil contamination, e.g., by smoke.

ROCK FRAGMENT DETERMINATION

There were no deviations from the specified procedures for rock fragment determination. Laboratory 4 reported that bulk samples containing rock fragments greater than 20 mm in diameter were brought in from the field for a few pedons. If this were the case, rock fragment data for these samples may have identified all rock fragments greater than 2 mm rather than only the 2- to 20-mm fraction.

SOIL HOMOGENIZATION

There were no deviations from the specified procedures for soil homogenization. Label B identifying each sample for the analytical laboratories was placed on the appropriate sample bag, and each label A was placed into the Label A log book according to protocol. Remaining sample material was stored as specified in the protocols.

QUALITATIVE TEST FOR INORGANIC CARBON

There were no deviations from the specified procedures for the inorganic carbon test.

MOISTURE DETERMINATION

The protocols do not specify a procedure for determining soil moisture content. Consequently, Laboratories 1, 2, and 3 used one method (Method 1), whereas Laboratory 4 used a slightly different approach (Method 2). Method 1 utilized two subsamples of soil. The first air-dry sample was oven-dried to determine its moisture content. On the following day or at least 24 hours later, a second air-dry sample went through the same procedure. Unless the absolute difference between the moisture contents of the two samples was less than 2.5 percent, the procedure was repeated. In contrast, Method 2 utilized only one soil sample. The air-dry sample was weighed one day, was reweighed the following day, and was oven-dried. Then the moisture content of the sample on the different days was determined.

Both methods were supposed to ensure that samples had reached an equilibrium air-dry state. If the absolute moisture content of a sample changed less than 2.5 percent in 24 hours, the soil was considered air-dry. This could be an erroneous assumption in a humid laboratory where, for example, a soil containing 20 percent moisture may dry slowly enough to remain within 2.5 percent of the initial measurement for 24 hours. By adhering to the methodology, the soil would be considered air dry and ready for further analyses when, in fact, it is not. However, when the moisture data were reviewed less than 3 percent of the soils had an air-dry moisture content greater than 10 percent.

A concern with Method 1 is the amount of soil used in the procedure. After a sample had been oven-dried, it could not be placed back into the bulk sample. If the moisture values of the initial two samples were not within 2.5 percent, a third sample was measured, and a comparison was made between samples two and three. This process continued until an equilibrium was reached, occasionally consuming a substantial amount of soil. Because the bulk sample was not homogenized before this determination and was discarded after measurement, the quantity of soil consumed adversely affected the representativeness of the sample and the determination of rock fragments in the bulk sample.

Another concern relates to moisture retention. An individual moisture sample will dehydrate at a faster rate than the bulk sample from which it is taken. The difference between the initial and second measurement, as used in Method 1, would better assess the air-dry moisture content of the bulk sample.

BULK DENSITY DETERMINATION

Clod analysis was chosen as the general method for determining bulk density, despite a few disadvantages. Obtaining clods from dry, loose, or extremely wet soils and from horizons containing many rock fragments can be difficult or impossible. Another concern is that the bulk density values obtained may be higher than the average bulk density of the soil horizon they represent, because sampling could be biased toward the collection of firmer, more compact clods capable of withstanding disturbance during sampling and transport.

Two very similar methods were used to determine bulk density. The methods varied because of the manner in which the volume of the clod was determined. The first method (Method 1) determines clod volume by placing a beaker of water on a balance, taring the balance, submerging the clod in the water, and using the weight of the submerged clod as a direct estimate of the volume of the clod. This method of determining volume is described in the protocols. The second method (Method 2) utilizes a stand and a beam that are placed on a balance and are tared. A clod is suspended from the beam and is allowed to submerge in water. The weight of the clod is recorded, and this value is subtracted from the air-dry clod weight in order to determine the volume of the clod.

The use of either Method 1 or 2 was based on each laboratory's familiarity with the method. Both methods are valid, although each method has its limitations. If a clod floats while Method 1 is being used, the weight of the floating clod does not reflect its true volume because some of the clod is above water. The advantage of this method is that the clod can be forcibly submerged, and its true volume can be recorded. If Method 2 is employed, the measured weight of the floating clod is zero. Using this method, bulk density values for floating clods would have to be removed from the data base because there is no alternative method for identifying their true volume.

Another common source of error associated with both methods was the failure to subtract the weight of the clod tag and hairnet surrounding the clod. Laboratory 1 attempted to compensate for the error by subtracting the oven-dry weight of the tag and hairnet from the oven-dry clod weight. The error surfaced during QA data evaluation, when it was observed that clods from the field occasionally weighed more than the same clods with additional laboratory coatings of saran. The major effect of this error occurs in the calculation of saran weight and density.

Variations in bulk density measurements also occur when clods are allowed to air dry between the initial and second weighing, i.e., after the clods have been coated with saran. Because water vapor can permeate saran, any loss of moisture could affect the recorded saran weight and density. If laboratory personnel initially weighed a moist clod from the field, dipped the clod a number of times, and allowed it to dry for several hours, the clod could lose an amount of moisture greater than the weight of the laboratory saran coatings. The effect on the bulk density value would be dependent upon the size and original moisture content of the clod.

Each preparation laboratory used a slightly different procedure for bulk density determination. The following paragraphs describe the actual bulk density procedure for each laboratory.

Laboratory 1

Method 2 was used to determine clod volume in water. An extra step was included in the calculation to subtract the oven-dry weight of the hairnet and tag that were attached to each clod.

Laboratory 2

Method 2 was used to determine clod volume in water. The laboratory underestimated bulk density values by failing to subtract the saran density from the bulk density measurement. For a number of raw data observations (187), a field clod weight and a clod weight after one saran coating were recorded. The remainder of the raw data observations (690) did not contain field clod weights. The bulk density, minus saran density, was calculated using the 187 data observations. Regression analyses that compared the 187 field clod weights with the corrected bulk density values produced an algorithm that could be used to calculate corrected bulk density values for the remaining data. The algorithm generated a strong correlation ($r^2 = 0.9987$) and was used to convert the bulk density values for the remaining 690 data observations. The average increase in bulk density due to the subtraction of saran was slight, i.e., 0.57 percent.

The relationship identified by the regression analyses is as follows:

$$Db = a(uDb) + b$$

where:

Db = corrected bulk density, minus saran
a = slope of the line (1.0413)
uDb = uncorrected bulk density, including saran
b = y-intercept (0.0438)

Laboratory 3

Method 2 was used to determine clod volume in water. While calculating the bulk density, the laboratory used an erroneous algorithm from the protocols. The saran volume was added to the clod volume in the denominator of the equation, rather than being subtracted from it. This discrepancy skewed each bulk density value an average of 4.7 percent. All values later were recalculated using raw data supplied by the laboratory.

Laboratory 4

Method 1 was used to determine clod volume in water. Because the clod tag and hairnet weights were not subtracted from the clod weight, there were instances of laboratory clod weights that were less than the field clod weights. The data for the 22 affected clods later were removed from the data base.

Because this laboratory used Method 1, an attempt was made to identify any floating clods. This method involves the use of a closed system, i.e., a floating clod should weigh the same amount in water as it does in air. The bulk density values for clods whose weight in water was within 10 grams of the air-dried weight were reviewed, as well as the values for clods weighing more in water than in air. It was concluded that the floating clods were forcibly submerged in order to record the true volume, and the laboratory manager confirmed this.

SAMPLE SHIPMENT

There were no deviations from the specified procedure for sample shipment. Samples were shipped from the preparation laboratories to the analytical laboratories via overnight air courier.

SECTION 4

QUALITY ASSURANCE/QUALITY CONTROL

A QA/QC program must be followed during the course of survey activities to ensure that the resulting data are of known quality. The QA/QC program for the Northeastern soil survey consisted of design and evaluation components that aided survey participants in obtaining data that meets the needs of the end users.

The QA/QC design for the preparation laboratories included the training of personnel in the protocols to be followed, establishment of a communications network, assessment of data quality objectives, and accomplishment of on-site systems audits. The data were systematically evaluated to ensure data quality, and QA samples were included in each batch of routine samples to facilitate this evaluation. The following sections explain some of these aspects of the QA/QC program in relation to preparation laboratory activities. Other aspects of the program will be addressed in the quality assurance report on the analytical laboratory data.

DESIGN COMPONENTS

Training

Preparation laboratory personnel attended a regional workshop held in Orono, Maine, on August 7 and 8, 1985. The purpose of the workshop was to review the laboratory protocols and to establish a consistent methodology for the laboratories that would ensure the comparability of data.

Communications

Weekly conference calls assisted in keeping the preparation laboratories operating efficiently and consistently by providing a forum that allowed potential difficulties to be identified, discussed, and resolved. Preparation laboratory managers, QA personnel, and scientists involved in the study of soil mineralogy participated in these calls. Issues discussed during the conference calls included supply shortages and clarification of procedures, e.g , sample labeling, record keeping, and drying of organic samples.

Data Quality Objectives

Data quality objectives for the preparation laboratories were not established before the Northeastern survey was conducted. The preparation laboratories were assessed according to the following data characteristics:

- Precision and Accuracy - These are quantitative measurements that estimate the amount of variability and bias inherent in a given data set. Precision refers to the level of agreement among repeated measurements of the same parameter. Accuracy refers to the difference between an estimate based on the analytical results and the true value of the parameter being measured.

- Representativeness - This refers to the degree to which the collected data accurately reflect the population or medium that is sampled.

- Completeness - This refers to the amount of data that is successfully collected with respect to the amount intended in the design. A defined percentage of the intended amount must be successfully collected for conclusions based on the data to be valid. Lack of data completeness may reduce the precision of estimates, may introduce bias, and may therefore reduce the level of confidence in the conclusions.

- Comparability - This refers to the similarity of data from different sources included in a single data set. If more than one laboratory is analyzing samples, uniform procdures must be used to ensure that the data from different sources are based on measurements of the same parameter.

These five data quality characteristics were identified in the DDRP QA Plan (Bartz et al., 1987), and their application to preparation laboratory activities were iterated. A brief description of the specific characteristics follows:

- Precision and Accuracy - The preparation laboratory combines sets of field samples into one batch containing a maximum of 39 routine and duplicate samples. After processing, i.e., air-drying, crushing, sieving, and homogenization, one bulk sample is split into two subsamples which are termed preparation duplicates. Comparison of physical and chemical data for these duplicates allows evaluation of the subsampling procedure.

- Representativeness - Each bulk sample is processed by a preparation laboratory to obtain a homogeneous sample. Homogenization is accomplished by passing the sample through a Jones-type riffle splitter at least seven times. The riffle splitter also is used for subsampling. All samples not being processed are stored at 4°C by the preparation laboratory.

- Completeness - Each batch of samples sent to a contractor analytical laboratory includes the preparation duplicates.

- Comparability - All preparation laboratories process bulk samples according to the protocols. Strict adherence to protocols should result in comparability among preparation laboratories.

Precision is identified in this QA report by evaluating field duplicate data from the rock fragment and inorganic carbon analyses and replicate data from the bulk density analyses. Additional precision estimates are made by evaluating analytical data from the preparation laboratory duplicates and will be documented in the QA report on the analytical laboratory data.

Accuracy cannot be assessed because: (1) true values for the parameters in question are not known, and (2) it was not known how to provide preparation laboratories with audit samples and quality control calibration samples to evaluate the rock fragment and bulk density analyses.

Representativeness of the subsampling procedure will be assessed in the QA report for the analytical data.

Completeness is evaluated in this report by assessing each laboratory's ability to accomplish the processing and analyses tasks for the number of routine and duplicate samples assigned.

Comparability will be evaluated by using the precision data from the preparation duplicates. This evaluation will be documented in the QA report on the analytical laboratory data.

On-Site Systems Audits

Each preparation laboratory was audited at least once by QA staff. A round of pre-sample audits was performed before sample processing was underway. The purpose of the pre-sample audit was to evaluate the facilities and equipment and to ensure the capacity and suitability of the laboratory to function as a preparation laboratory for the Northeastern soil survey. The same QA auditor performed all pre-sample audits, which ensured a consistency of observation and inquiry. Issues not treated in the protocols, e.g., log books and deviations from written methods, were discussed and were resolved. An on-site evaluation form was required to be filed together with the pre-sample audit report. This evaluation form contained specific questions relating to the type of equipment available, safety procedures, and methodology.

The pre-sample auditor concluded that all preparation laboratories selected for the Northeastern soil survey either had or could obtain adequate facilities and equipment to perform the functions specified in the protocols. The following comments highlight specific concerns of the auditor:

- The soil drying room of Laboratory 2 was located on the seventh floor of an office building, whereas the other laboratories used greenhouses. The auditor was concerned about inadequate drying space as well as proper ventilation for efficient drying. The laboratory planned on adding extra shelving.

- The existence of an ammonia leak in the cold storage area of Laboratory 3 was identified. A test to determine whether the leakage would contaminate the soils was proposed (see Section 3). All samples were double-bagged in order to decrease the likelihood of contamination.

A second round of audits was performed on Laboratories 3 and 4 while the laboratories were preparing samples. The QA auditor evaluated their procedures and followed up on concerns identified in the pre-sample audit. The auditor found that the laboratories were operating in adherence to protocol, and the only area of concern identified was the need for better record-keeping at Laboratory 3. An ERL-C representative performed systems audits at Laboratories 1 and 2 during the soil preparation activities, however written audit reports documenting the visits are unavailable.

DATA EVALUATION

Quality Assurance Samples

Three types of paired QA samples were included in each batch of samples submitted to the analytical laboratory: (1) field duplicates, (2) preparation duplicates, and (3) audit samples.

One horizon per sampling crew per day was sampled in duplicate as specified in the protocols. The first of the duplicate pair was considered the routine sample, and the second of the pair was referred to as the field duplicate. The field duplicate underwent the same preparation steps as its associated routine sample in order to allow an estimate to be made of sampling and horizon variability.

One sample per batch was chosen by the preparation laboratory to be homogenized and split into two subsamples. The preparation duplicates were designed to allow a quantitative estimate of physical and chemical variability in splits of the sample material. Comparing data from analyses of preparation duplicates allows a reliable estimate to be made of how well the soil samples were homogenized before being subsampled. An evaluation of preparation duplicate data will be presented in the QA report on the analytical laboratory data.

Two natural audit sample pairs supplied by EMSL-LV were included in each batch that was sent to an analytical laboratory via a preparation laboratory, but the audit samples did not undergo any processing at the preparation laboratory. These samples are used to assess analytical performance. More information on these audit samples can be found in the QA plan (Bartz et al., 1987). An evaluation of the audit sample data will be presented in the QA report on the analytical data.

Method of Estimating Analytical Precision

Data for the bulk density and fock fragment determinations were grouped into data sets, by laboratory. A scatter plot of horizon standard deviation versus horizon mean was generated from each data set to evaluate the relationship between precision and concentration. The data for both parameters displayed a random pattern of standard deviation, indicating that precision was independent of concentration. On this basis, a completely randomized design model was selected for the statistical estimation of precision (Steel and Torrie, 1960). The model that represents data collected at a specific sampling site can be demonstrated as follows:

$$y_{ij} = u + h_i + e_{ij}$$

where:

y_{ij} = the variable of interest for the jth number of observations from the ith number of individual horizons represented,

u = the general mean of the population,

h_i = the effect of the ith horizon on the variable of interest,

e_{ij} = the random error of analytical measurement for the jth number of observations from the ith number of individual horizons represented.

The model was used to perform statistical analyses of (1) bulk density data from the clod replicates and (2) rock fragment data from the field duplicates. A root mean square error statistic was used to estimate the pooled standard deviation (S_p) across all pedons and horizons for each laboratory data set, and the coefficient of variation (CV) was derived by dividing the S_p by the general mean ($\overline{X}$) and multiplying by 100.

PRECISION RESULTS FOR BULK DENSITY DETERMINATION

Data from the bulk density determination were analyzed by the completely randomized design model to provide overall S_p and CV values for the sets of replicate clods at each preparation laboratory. In addition, CV values were generated for sets having a within-set mean bulk density that was either greater than or less than the general mean bulk density. Summary statistics for the S_p and CV values are given in Table 1.

The CV values less than and greater than the mean were evaluated in order to determine whether or not the statistical relationship of higher CV values at lower concentrations, in this case at lower bulk densities, would hold true for bulk density data. The CV values presented in Table 1 appear to confirm this relationship.

TABLE 1. PRECISION ESTIMATES FOR BULK DENSITY

	Number of Horizons Sampled	Mean Bulk Density (g/cm^3)	S_p	CV	CV $<\overline{X}$	CV $>\overline{X}$
Laboratory 1	235	1.24	0.110	8.82	10.52	7.66
Laboratory 2	303	1.38	0.096	6.97	9.85	5.26
Laboratory 3	50	1.61	0.178	10.99	13.50	8.49
Laboratory 4	251	1.35	0.087	6.48	7.54	5.79

The S_p and CV values for the Laboratory 3 replicates suggest a higher imprecision than values for the other laboratories. The mean bulk density of samples analyzed by Laboratory 3 also is higher than that of the other laboratories. Because they finished sampling earlier in the season when the weather was warm and dry, the sampling crews supplying Laboratory 3 may have had difficulty collecting clods from surface horizons. Soil conditions may have favored the collection of clods from the lower, moister horizons. The collection of replicates may not have been as representative of a horizon as desired.

The overall S_p values suggest a consistency of bulk density values within a horizon. Audit reports indicated that the sampling crews were able to choose representative clods from each horizon and that the laboratories were consistent in their use of measurement techniques. However, the exact percentage of error contributed to the S_p by horizon variability, sampling bias, or laboratory imprecision cannot be determined because (1) inherent spatial variability made it impossible to sample identical field clods or to provide an audit sample for estimation of potential sampling bias, and (2) the preparation laboratories were not provided with calibration samples to allow estimation of laboratory bias.

The following types of sampling errors could contribute to sampling bias for the bulk density replicate clods:

- Collection of replicates from transitional zones or adjacent horizons
- Mislabeling or careless handling of clods
- Inconsistent saran coating procedure
- Bias relating to the structural coherency of clods

Based on the experience of the Soil Conservation Service (SCS) sampling crews and the fact that the various field audit reports did not indicate any major deviations from the protocols, sampling bias is not presumed to have been a significant factor affecting the S_p values. Some variability in the use of saran, e.g., the length of time clods were allowed to be submerged in saran, was mentioned in a few audit reports. The S_p values would not be affected if the coating procedure was consistent for all replicates from a horizon.

At the preparation laboratory, measurement or method errors can introduce bias, including:

- Transcription erors in recording clod weight or sample code
- Inconsistent saran coating procedures
- Improper clod handling, e.g., compaction
- Incomplete drying
- Loss of soil material during sieving

- Inaccurate calculations

- Faulty weights, e.g., clod tags and hairnets not subtracted

Because audit samples and calibration samples were not provided, laboratory bias could not be measured. Therefore, it is difficult to quantify the potential effect of preparation laboratory bias on the S_p and CV values.

PRECISION RESULTS FOR ROCK FRAGMENT DETERMINATION

Field duplicate data for the rock fragment determination were analyzed by the completely randomized design model to provide overall S_p and CV values for each preparation laboratory. Summary statistics incorporating these values are provided in Table 2.

TABLE 2. PRECISION ESTIMATES FOR ROCK FRAGMENTS

	Field Duplicate Pairs	Mean % Rock Fragments	S_p	CV
Laboratory 1	64	25.1	4.002	15.97
Laboratory 2	76	16.2	1.792	11.04
Laboratory 3	21	14.2	3.367	23.70
Laboratory 4	62	12.0	1.135	9.47

Because of the simplicity of the method used for determining the percentage of rock fragments in the bulk soil samples, a greater amount of imprecision can be attributed to a combination of sampling bias and horizon variability and a lesser amount to preparation laboratory bias. The imprecision may be an indication of improper field duplicate sampling technique or considerable spatial variability in rock fragments.

COMPLETENESS RESULTS

The requested analyses and soil processing steps were performed on 100 percent of the bulk samples and clods received by the preparation laboratorries, which satisfied the theoretical maximum design level of completeness. Preparation duplicates were created for each batch of samples sent to the analytical laboratories, for a 100 percent level of completeness.

SECTION 5

CONCLUSIONS AND RECOMMENDATIONS

The conclusions and recommendations discussed below are summarized from discussions at the exit meeting, conference call notes, and the examination of raw data.

DATA RECORDING

Verifying the data from the Northeastern soil survey was difficult because the format of log books in which the data were recorded was not consistent from one laboratory to another. A system has been designed that would allow the raw data to be entered into log books containing pre-printed data forms, followed by data entry into a computer data set having the same format. This would facilitate the verification process and would relieve preparation laboratory personnel of calculating final values as well. Possible components of a data recording system are discussed below.

Preparation Laboratory Forms

Standardized forms should be developed on which to record data from all preparation laboratory procedures. Recommended formats are presented for the following forms:

- Figure 1 - Sample receipt
- Figure 2 - Bulk density
- Figure 3 - Rock fragment
- Figure 4 - Inorganic carbon
- Figure 5 - Percent moisture
- Figure 6 - Sample processing

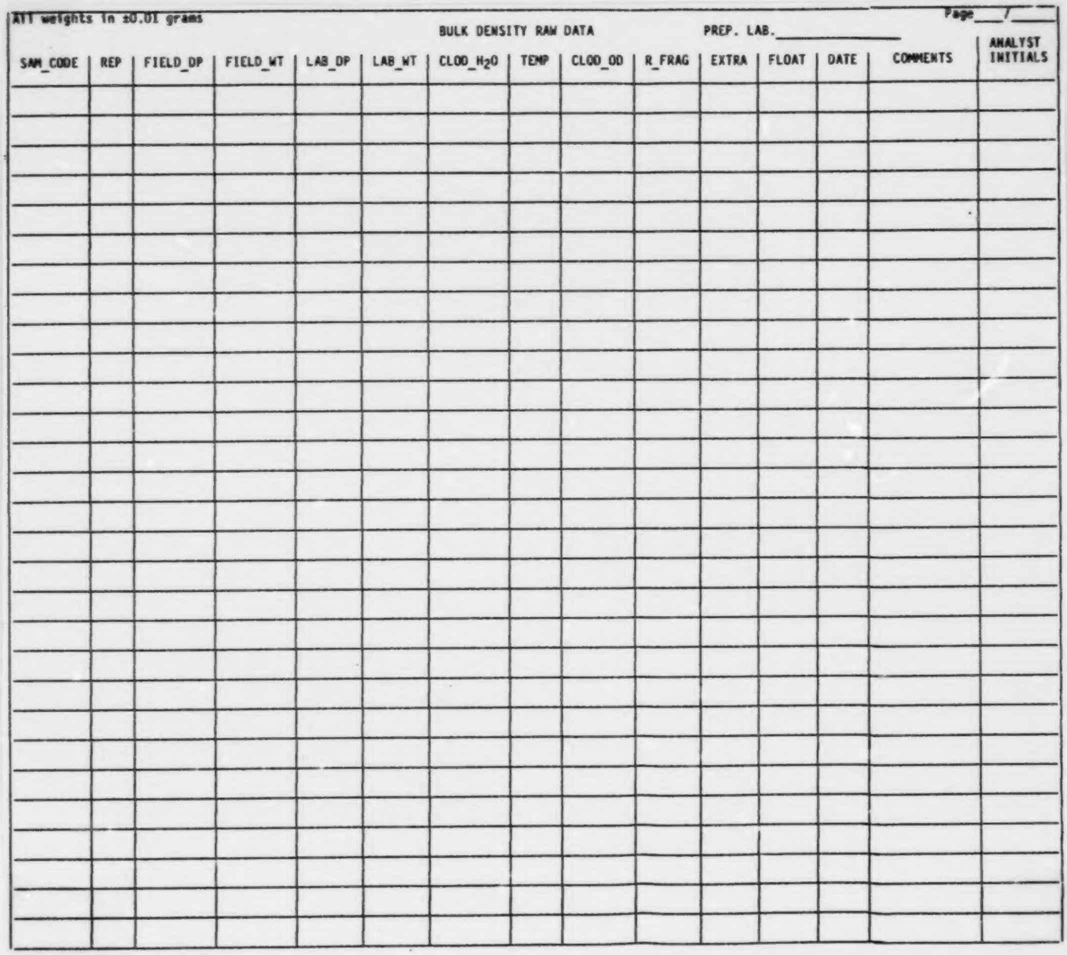
Note that most of the forms use data-file variable names as the column headings.

Data Entry Procedures

Preparation laboratory personnel will not be required to calculate final values, so there will be no entry field for final values on the raw data forms.

Sample Number	Crew ID	Site ID	Date Collected	Date Received	Time Received	Delivered By	Received By	Sample Condition	Additional Notes		
								Wet/Dry (W/D) Sieved/Unsieved (S/U) Bag Split (BS) Under Volume (UV)	Number of Clod Samples Collected for Each Horizon Sampled	Field Duplicate	Paired Pedon

Figure 1. Sample receipt form.



All weights in ±0.01 grams

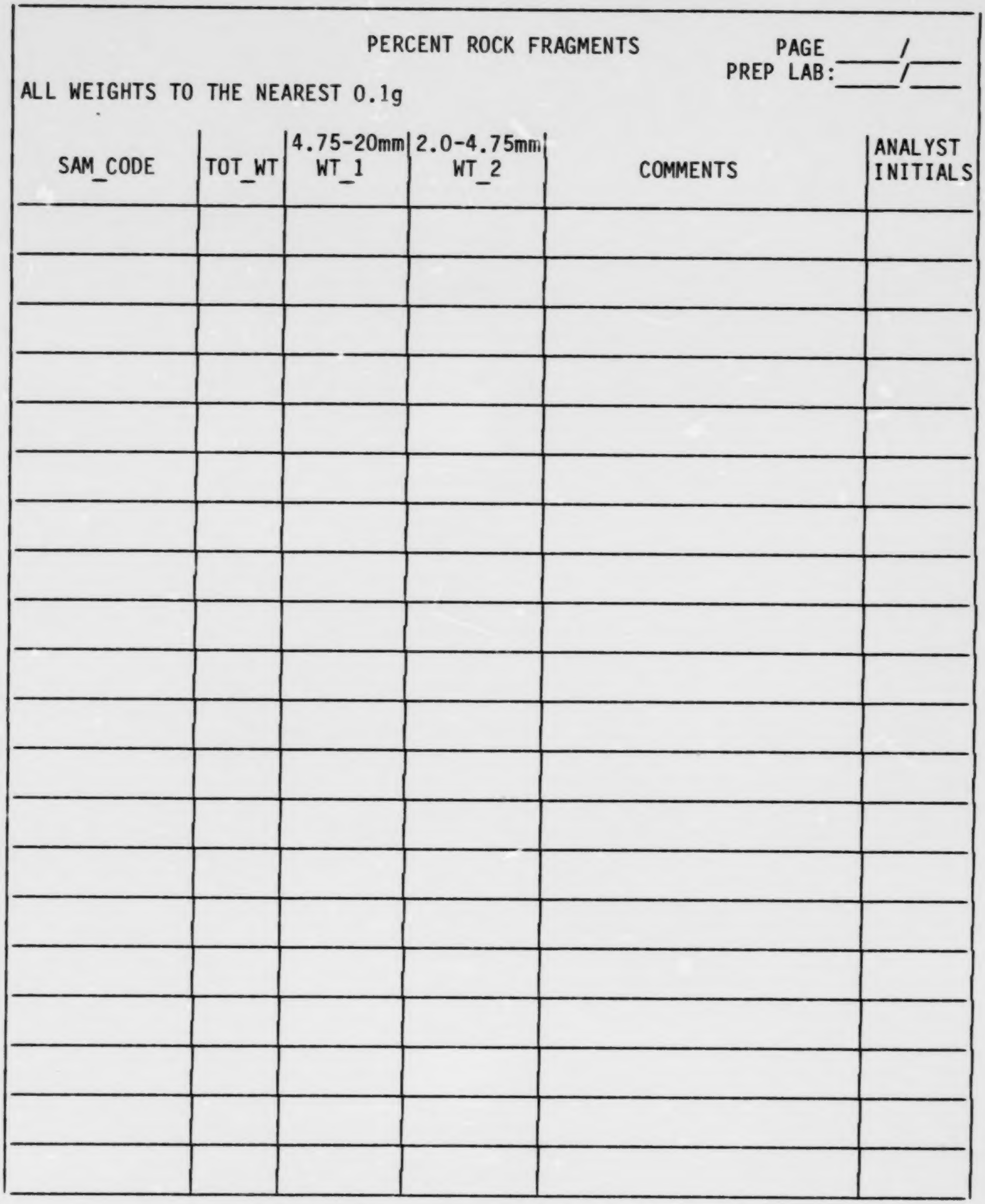
BULK DENSITY RAW DATA

PREP. LAB.________________

Page___/_____

SAM_CODE	REP	FIELD_DP	FIELD_WT	LAB_DP	LAB_WT	CLOD_H_2O	TEMP	CLOD_OD	R_FRAG	EXTRA	FLOAT	DATE	COMMENTS	ANALYST INITIALS

Figure 2. Bulk density raw data form.



PERCENT ROCK FRAGMENTS

PAGE ____/____

PREP LAB:____/____

ALL WEIGHTS TO THE NEAREST 0.1g

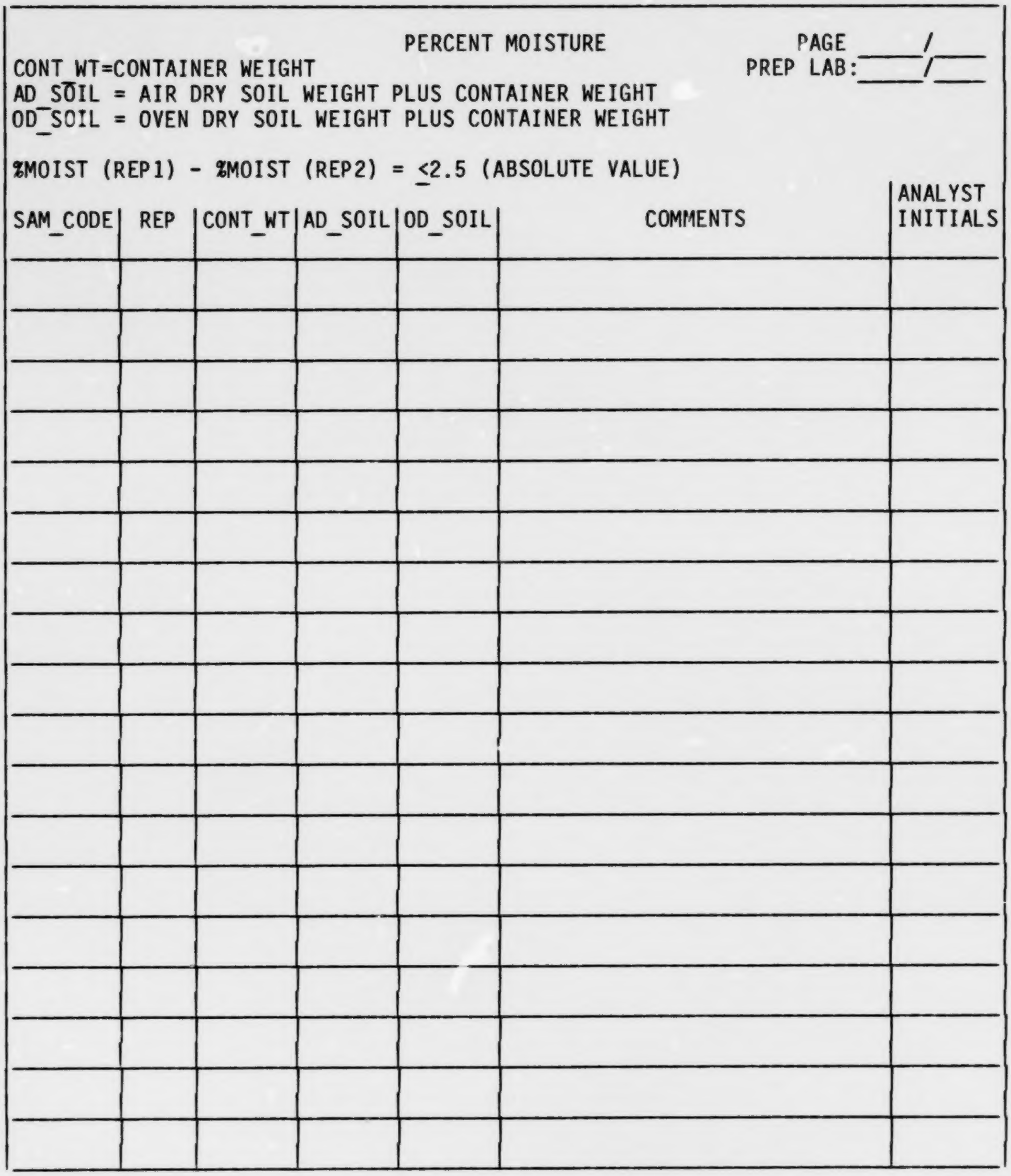
SAM_CODE	TOT_WT	4.75-20mm WT_1	2.0-4.75mm WT_2	COMMENTS	ANALYST INITIALS

Figure 3. Rock fragment raw data form.

TEST FOR INORGANIC CARBON PAGE ____/____
PREP LAB:____/____

SAM_CODE	REP	YES	NO	COMMENTS	ANALYST INITIALS

Figure 4. Inorganic carbon raw data form.



PERCENT MOISTURE

PAGE _____/____

CONT_WT=CONTAINER WEIGHT

PREP LAB:_____/____

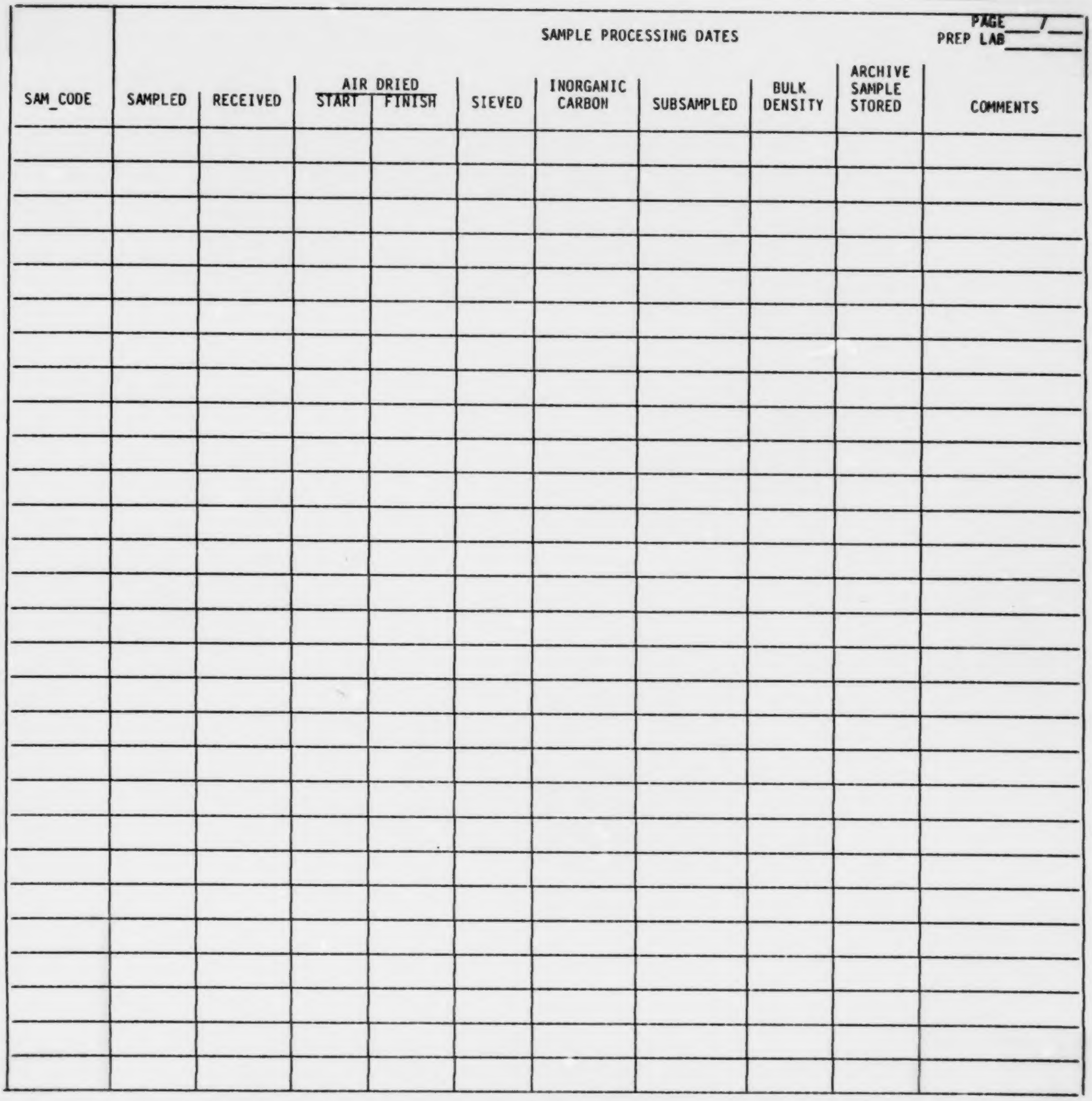
AD_SOIL = AIR DRY SOIL WEIGHT PLUS CONTAINER WEIGHT

OD_SOIL = OVEN DRY SOIL WEIGHT PLUS CONTAINER WEIGHT

%MOIST (REP1) - %MOIST (REP2) = $\leq$2.5 (ABSOLUTE VALUE)

SAM_CODE	REP	CONT_WT	AD_SOIL	OD_SOIL	COMMENTS	ANALYST INITIALS

Figure 5. Percent moisture raw data form.



PAGE ___/___
PREP LAB ______

	SAMPLE PROCESSING DATES									
			AIR DRIED							
SAM_CODE	SAMPLED	RECEIVED	START	FINISH	SIEVED	INORGANIC CARBON	SUBSAMPLED	BULK DENSITY	ARCHIVE SAMPLE STORED	COMMENTS

Figure 6. Sample processing form.

However, the preparation laboratory supervisor should calculate bulk density from random samples in order to ensure placement of the variables into the correct fields. Soil samples do not have to be analyzed in the order they appear in the batch because the sample code information linking the data to a master form will be placed on each raw data form.

On a weekly basis, the preparation laboratory personnel will enter all raw data into a data base file by using dBase III software. The program format will have data entry screens that display facsimiles of the raw data forms. If a preparation laboratory does not have access to a computer system, QA personnel will enter the data. After data are entered, either of two options exist:

- Option 1 - The software can be programmed to calculate final data. The calculated final data can be generated on the Form 101 under the correct sample code, and the preparation laboratory can access and print these data. Algorithms to calculate percent relative standard deviations for field duplicates, routine samples, and the bulk density replicates can be incorporated into the software. Outliers, erroneous values, and invalid sample code entries can be identified at this stage.

- Option 2 - Preparation laboratory personnel can enter the raw data. No statistical calculations or conversions of raw data to final data would occur at the preparation laboratories. Instead, the raw data would be sent electronically to the QA staff. QA personnel can convert the dBase III data into a SAS data set and can calculate final values. Next the data can be analyzed statistically, and the findings reported to the laboratories.

Either option should work equally well. Data entry errors can be identified before batches are sent to the analytical laboratories. In fact, data verification may be completed shortly after the final batch of soil samples is assembled at the preparation laboratory.

The sample processing form will provide a record of the sample codes from the master form and the date of specific laboratory analyses from the raw data forms. The sample processing form can verify that all required analyses were performed on the samples.

SAMPLE DRYING

Certain procedural details concerning sample drying were inadequately addressed in the protocols. The procedure should be expanded as follows:

- Drying efficiency would improve if the samples were stirred every 24 hours to facilitate uniform drying. Steel mesh trays covered with kraft paper could be used for sample drying. This would eliminate the water condensation that occurred with the cafeteria-type trays.

- Specifications for drying facilities, e.g., temperature and humidity controls, space allocation, access, and air circulation, should be stipulated.

- Special procedures for handling certain soil types, e.g., organic or clayey soils, should be provided.
- Soils that are known to harden upon drying, e.g., mucky or clayey soils should be crushed and mixed before they reach constant moisture content.
- When handling the samples, the analyst should wear rubber or plastic gloves to lessen the possibility of contamination.
- Elimination of all sources of contamination, e.g., smoking and food, should be stipulated.

MOISTURE DETERMINATION

Analysis of the initial raw data values for percent moisture suggests that the preparation laboratory personnel were capable of assessing the air-dry condition of samples. The following protocol modifications should be made for the moisture determination:

- The bulk sample should be allowed to dry until the preparation laboratory personnel judge the sample to be air dry.
- A 10-gram air-dry subsample can be removed for determination of percent moisture.
- A determination of field moisture should be made on all samples upon their arrival at the preparation laboratories. A consistent method for measuring field moisture must be added to the protocols.

BULK DENSITY DETERMINATION

The following recommendations are made to alleviate difficulties with the bulk density procedure.

- An audit sample should be included to provide data for estimating within- and among-laboratory precision and interlaboratory bias. (See page 30).
- A mineral of known density, e.g., quartz, should be used as a quality control calibration sample. Because the data have demonstrated that measurement precision is not dependent upon the level of bulk density, it is not necessary for the quality control calibration sample to have a bulk density within the range of soil bulk density values.
- Clods should air-dry before processing for the bulk density determination. Clod weight should be recorded immediately before and soon after laboratory saran coating.
- Method 1, which was outlined in the protocols, is the preferred method for determining bulk density. The method should be rewritten to correct erroneous algorithms (see Section 2, pp. 5 and 6) and to add specific instructions for submerging clods.

- Floating clods should be identified on the bulk density raw data form.
- Clod tags should be removed or should be dried before the clods are weighed.
- Because saran and acetone are carcinogenic, laboratory managers should take extra care to ensure that saran mixing, clod coating, and clod ignition are performed in an operating exhaust hood. Operators should wear respirators.

QUALITY ASSURANCE/QUALITY CONTROL

Communications

Although the conference calls met the objective of establishing a forum where questions could be answered or issues could be identified and resolved quickly, documentation of the discussions and dissemination of the information were not done consistently. The following suggestions are made:

- The QA auditor should lead all conference calls. This person or another member of the QA staff will be present for all conference calls.
- The frequency of conferences should be weekly, unless the QA auditor decides that biweekly calls would be equally effective.
- All pertinent information should be documented by the conference leader in a log book established specifically for the soil survey region. This book can identify the participants involved in the discussions as well as key personnel not in attendance.
- Log book notes should be compiled and should be sent to the appropriate participants weekly.

Data Quality Objectives

Data quality objectives should be established for the determinations of bulk density and percent rock fragments.

On-Site Systems Audits

The pre-sample audit achieved the objective of assessing the ability of each laboratory to function as a DDRP preparation laboratory. The second audit was instrumental in evaluating adherence to protocol during the soil processing. Each laboratory should be evaluated before and during sample processing. It is recommended that one QA auditor should evaluate all the laboratories, for both the pre-sample and the second audit. If this cannot be accomplished, one auditor should perform all pre-sample audits, and another, the second evaluations. This would facilitate a consistent assessment of all laboratories.

Audit Samples

Audit samples are needed to evaluate preparation laboratory accuracy for the bulk density determination. The following has been suggested, but has not been tested at EMSL-LV. QA personnel could provide the preparation laboratories with synthetic samples, e.g., plastic eggs filled with materials of known densities. The eggs would be coded, and the bulk density of each egg would be determined at the QA laboratory. A set of 24 eggs could be sent to each preparation laboratory, which would be required to calculate the bulk density of one egg for each day the laboratory was analyzing clods.

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APPENDIX A

SAMPLING AND PREPARATION LABORATORY PROTOCOLS FOR THE DIRECT/DELAYED RESPONSE PROJECT SOIL SURVEY

The following protocols were used by the field crews and the preparation laboratory personnel participating in the Northeastern DDRP Soil Survey. The draft manual was revised using the information obtained from the sampling and preparation laboratory training workshop held on August 7 and 8, 1985. The draft did not undergo external review and was not formally released by EPA. It is presented here without editorial correction. Note that various Soil Conservation Service documents were used in the preparation of this draft; however, because no editorial corrections have been made, those documents are not cited.

FIELD SAMPLING MANUAL FOR THE NATIONAL ACID DEPOSITION SOIL SURVEY

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The mention of trade names or commercial products in this manual is for illustration purposes, and does not constitute endorsement or recommendation for use.

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1.0 INTRODUCTION

1.1 Scope

This field sampling manual is written to guide personnel involved in the collection of soil samples for the U.S. Environmental Protection Agency's (EPA) Direct/Delayed Response Project (DDRP) Soil Survey. All field and laboratory personnel must be trained by a field manager or another person knowledgeable in the procedure and protocols detailed in this manual. The scope of the field sampling manual covers field operations, shipping of samples from the preparation laboratory to the analytical laboratory, and sample receipt by the analytical laboratory.

This manual is a companion to the [laboratory] Methods Manual for the National Acid Deposition Soil Survey (NADSS) and the Quality Assurance Plan for the National Acid Deposition Soil Survey (NADSS). There is some repetition among the manuals which is necessary to maintain continuity and to document concisely the methodology of the soil survey.

The basic goals of the National Acid Deposition Soil Survey (NADSS) procedures are to collect representative samples without contamination, to preserve sample integrity for analysis, and to analyze samples correctly. Analytical methods have been chosen that offer the best balance between precision, accuracy, sensitivity, and the needs of the data user.

The overall objective of NADSS is to predict the long-term response of watersheds and surface waters to acidic deposition. Based on this research, each watershed system will be classified according to the time scale in which it will reach an acidic steady state, given current levels of deposition. Three classes of watershed systems are defined:

> Direct response systems: Watersheds with surface waters that either are presently acidic (alkalinity <0), or will become acidic within a few (3 to 4) mean water residence times (<10 years). NOTE: Most lakes in the Northeast have relatively short residence times, i.e., less than 2 to 4 years.
>
> Delayed response systems: Watersheds in which surface waters will become acidic in the time frame of a few mean residence times to several decades (10 to 100 years).
>
> Capacity protected systems: Watersheds in which surface waters will not become acidic for centuries to millennia.

The objectives of this manual are to define the means by which to characterize and sample soil mapping units using U.S. Department of Agriculture, Soil Conservation Service (USDA-SCS) descriptive techniques.

1.2 Personnel

1.2.1 Field Sampling Crews - The field sampling crews will consist of soil scientists experienced in the National Cooperative Soil Survey. Crews will be numbered consecutively beginning with 01. For example, if Maine has three crews, they will be ME01, ME02, ME03. These crews will be responsible for selecting pedon location, sampling the soil, and describing the profile. The field crew leader will have ultimate responsibility for each crew's daily activities, such as placement of the pedon within each sample class, correct labeling of sample bags and forms, and prompt shipment of samples.

1.2.2 Regional Coordinator/Correlator - The Regional Coordinator/Correlator (RCC) will monitor six to ten percent of the sampling units to ensure adherence to SCS standards and field sampling protocol. Three to five percent of the sites will be monitored in conjunction with the monitoring responsibilities of the SCS staff of each state. The remaining sites will be monitored independently of the state SCS staff. Monitoring will include a review of profile descriptions and selection of sites for sampling. The RCC will be a qualified soil correlator with many years of experience with soil profile description and soil mapping. He will also ensure that the SCS State Office Staff perform duplicate profile descriptions. In this process, he will review these descriptions and point out potential problems.

1.2.3 Quality Assurance/Quality Control Representative - The Quality Assurance/Quality Control (QA/QC) representative will review five percent of the sampling units to ensure adherence to sampling protocol as specified in this manual.

1.2.4 SCS State Office Staff - Members of the SCS State Office Staff will independently describe five to ten percent of the sample pedons and site description and will monitor field sampling protocol. At least one site per state will be audited by the RCC representative. The use of duplicate profiles, determined prior to sampling, will assess variability in site description and sampling techniques between soil scientists and will check adequacy of site selection and labeling. This process requires that the staff perform their assessment while the crew is describing and sampling the pedons. NOTE: Reviews by the RCC, QA/QC Representative, and the SCS State Office Staff should be documented and all reports should be submitted to the EPA-Las Vegas QA Manager.

1.2.5 Soil Preparation Laboratory - Four soil preparation laboratories will participate in NADSS. These laboratories include the Cornell University Characterization Laboratory at Ithaca, New York, the University of Maine Soils Laboratory at Orono, Maine, the University of Connecticut Soil Testing Laboratory at Storrs, Connecticut, and the University of Massachusetts Soil Testing Laboratory at Amherst, Massachusetts.

Sample bags, data forms, labels, audit samples, shipping containers, and other equipment will be shipped to these soil preparation laboratories by EPA-Las Vegas. The field soil scientists will use these laboratories as sample drop-off points and supply pick-up points.

1.2.6 Analytical Laboratories - Routine and QA samples will be shipped in batches to each analytical laboratory from the preparation laboratory. Each batch will consist of a maximum of 39 routine samples and field duplicates, 2 audit samples, and a preparation laboratory duplicate.

2.0 SITE SELECTION

2.1 Watershed Selection

Because the objectives of DDRP are focused on making regional inferences, it was critical that the 150 watersheds selected for mapping of soils and watershed characteristics constituted a representative sample of the region. The 773 watersheds included in Region I of the National Surface Water Survey (NSWS) provided an excellent starting point from which to draw a subsample of 150 for the northeastern portion of DDRP, because (1) the NSWS lakes were selected according to rigorous probability sampling method (stratified by five subregions and three alkalinity classes within each subregion) and (2) water-chemistry information was available from NSWS for these lakes.

The 150 watersheds to be studied in DDRP also are part of the Phase II lake-monitoring program of NSWS that will provide a data set that contains both water-chemistry and watershed information. Therefore, the procedure used to select these watersheds incorporated criteria relevant to both DDRP and NSWS. The procedure consisted of five steps, which are summarized as follows:

Step 1: Lakes of low interest (too shallow, highly enriched, capacity protected, polluted by local activities, or physically disturbed) were excluded.

Step 2: Lakes too large to be sampled (>200 ha) were excluded.

Step 3: A cluster analysis was performed on a set of chemical and physical variables to group the remaining 510 lakes into three clusters of lakes with similar characteristics.

Step 4: A subsample of 60 lakes was selected from each cluster, then the three subsamples were weighted to represent the overall population of lakes in the Northeast.

Step 5: Lakes with watersheds too large to be mapped at the required level of detail (watersheds >300 ha) were excluded from the subsamples.

This procedure identified 148 lakes and watersheds, spread across the three clusters. Note that the three groups differ primarily in their alkalinities, pH levels, and calcium concentrations. To maintain the ability to regionalize conclusions drawn on the sample of 148 watersheds, the precision of information characterizing each of these watersheds should be comparable, and each cluster should be described at the same level of detail as the others.

2.2 Watershed Mapping

During the spring and summer of 1985, 145 of the 148 watersheds were mapped. The logistics and protocols of the watershed mapping are described in Chapters 6 and 7, Volume 5, Appendix B.2 Soil Survey -- Action Plan/ Implementation Protocol.

A total of about 440 mapping units were identified in the 150 watersheds. Sampling each of the 440 mapping units would not necessarily be the best way to describe adequately the chemistry of the region's soils. A better procedure is to combine the identified soils into groups, or sampling classes, which are either known or expected to have similar soil-chemical characteristics. Each of these sampling classes can then be sampled across a number of watersheds in which they occur, and the mean characteristics of the sampling class can be computed. These mean values and the variance about the mean can then be used to "build back up" area- or volume-weighted estimates of each watershed's characteristics.

For this procedure to work, it is critical that a sufficient number of samples are taken (five or more) to characterize the variability of each sampling class. This necessitates aggregating the number of mapping units into a reasonable number of sampling classes, given budgetary constraints. Thus, the central goal is to develop a method of grouping the large number of soils into a reasonable number of sampling classes.

2.3 Sampling Classes

2.3.1 Data Base - The data base contains about 2200 observations that were recorded on the field forms during the soil mapping of 145 watersheds selected as part of the DDRP and the Phase II lakes survey. This information includes:

Taxonomic class (series, subgroup, great group)
Parent material
- Origin
- Mode of deposition

Drainage class
Slope class
Slope configuration
Family texture
Geomorphic position
Dominant landform
Surface stoniness
Percent inclusions
Percent complexes
Estimated depth to bedrock
Estimated depth to permeable material

This information was considered in aggregating similar mapping units into sampling classes. The data base also includes the area of each mapping unit, number of occurrences, and percent of the watershed area.

Separate data files also exist for vegetation type, vegetation class, and geology. The data management system, dBase III, runs on an IBM PC-XT microcomputer at the EPA Environmental Research Laboratory in Corvallis, Oregon (ERL-C).

2.3.2 Evaluation of Sampling Classes - A taxonomic approach was used to identify 38 sampling classes as a foundation for aggregating similar mapping units. Taxonomic classification is based on similarities among soil properties. This taxonomic scheme was modified to reflect the major factors influencing soil chemistry.

2.4 Watershed and Sampling Class Selection

2.4.1 Sampling Class Objectives - The primary goal of this part of the sample selection procedure is to determine which sampling classes will be sampled in which watersheds. The sample sites should be selected to meet the following objectives:

Objective 1: To characterize all the sampling classes with similar levels of precision.

Objective 2: To describe the variation in watershed characteristics.

Objective 3: To describe the variation in the Acid Neutralizing Capacity (ANC) clusters developed from the lake survey.

2.4.2 Sampling Class Constraints - To meet these three objectives, a series of constraints was developed based on the allocation of samples to sampling classes and watersheds. The constraints that must be met follow:

Constraint 1: Approximately equal numbers of samples will be taken from each sampling class.

Constraint 2: Approximately two samples will be taken from each watershed.

Constraint 3: Not more than one sample will be taken from each sampling class in each watershed.

Constraint 4: Samples will be selected over the range of ANC clusters within each sampling class.

The method outlined here was developed to randomly select watersheds and sampling classes, within these constraints, using a simple selection algorithm.

2.4.3 Selection Algorithm - The method selection proceeds through a series of stages. Wherever possible, the rationale for the particular approach taken is described and cross-referenced with the objectives and constraints.

The selection method is based on the use of a systematic, weighted random sample of the watersheds that contain any given sampling class. First, number of samples to be taken in each sampling class is determined (constraint 1).

2.4.3.1 The first task is to construct a matrix of the occurrences of each sampling class in each watershed. This matrix is used to: (1) prepare a list of the watersheds that contain each sampling class, and (2) determine the number of different sampling classes in each watershed.

When the number of watersheds represented in each sampling class has been determined, it is possible to allocate the samples to sampling classes (given constraint 3).

Using eight samples per sampling class as a base, the following sample allocation occurs. Eight samples will be allocated to each sampling class where there are more than eight watersheds; where there are eight watersheds or less, one sample will be allocated to each watershed.

2.4.3.2 The next task is to determine which watersheds will be selected within each sampling class. In this process, constraints 2 and 4 are centrally important.

If watersheds are selected randomly within each sampling class, the watersheds that contain a large number of sampling classes will have more samples allocated to them than will the watersheds that have few sampling classes. To counteract this effect, and to help approach an approximately equal number of samples per watershed, the watersheds will be weighted (during the random selection procedure) by the inverse of the number of sampling classes that they contain.

For example, if one watershed contains four different sampling classes, it will be exposed to the sample selection procedure four times. Thus, it will be given one quarter of the weight of a watershed that contains only one sampling class. Using this technique, both watersheds have an approximately equal probability of being selected. This scheme will work accurately if there are equal numbers of watersheds considered in each sampling class; the presence of unequal numbers will cause some deviation from the most desirable distribution of samples.

To avoid overemphasizing the very common soils, only one sample will be taken from each watershed that contains only one sampling class.

All named soils in a complex soil series are counted as occurrences in their respective sampling classes. For example, a Tunbridge-Lyman soil complex in a watershed mapping unit would be considered as one occurrence of sampling class S12, which contains the Tunbridge series, and one occurrence of sampling class S13, which contains the Lyman series.

The method used to select watersheds within sampling classes will be to sort the watersheds by ANC cluster and then take a systematic, weighted random sample using the weights described above. This procedure selects a random starting point in the list of watersheds and then selects watersheds at regular intervals from the (weighted) list. This method ensures a selection across the range of ANC clusters.

To ensure that a watershed is not sampled more than once for a given sampling class, the weight assigned should not be larger than the interval used in the systematic sampling. Weights should be scaled down if they exceed the systematic sampling interval.

2.4.3.3 Once this procedure has been followed for each sampling class, the initial selection of watersheds and sampling classes can be summarized. Three options are possible at this point:

> The weighting factors can be adjusted iteratively until the allocation is acceptable.

Samples can be arbitrarily moved among watersheds to reach the desired allocation.

The selection can be accepted as adequate.

If the selection is not considered adequate, the most acceptable solution is to repeat the procedure using adjusted weights. This process could be automated, if necessary, with the weight of a watershed being increased until it receives sufficient samples.

The method of sampling class and watershed selection outlined here is designed to satisfy the objectives and constraints listed in Sections 2.4.1 and 2.4.2. Given the nature of the constraints, it is likely that there is no single, perfect solution; however, this method allows the production of an acceptable selection that is a compromise between the demands of the different objectives.

2.5 Final of Sampling Locations

2.5.1 Rationale and Objectives - Soil surveys generally have a holotypic purpose of describing the typical soil series or soil phases found in a watershed. The DDRP is interested in obtaining samples that are integrative or that represent the sampling class in the watershed. This sampling class may contain six or seven similar soils. The sampling purpose is not to describe the characteristics of a specific soil phase, but rather to describe the characteristics of the sampling class. Because all soils within a sampling class are considered similar in soil chemistry, the specific sampling location within a sampling class can be selected at random with respect to the soil series. The procedures described in this section are intended to (1) characterize the range of variability that occurs within a sampling class, and (2) characterize the soils within a sampling class using similar levels of precision.

Determining the sampling location within the watershed sampling class is a two-step process.

2.5.2 Sampling Site Selection - There are five steps in selecting representative sampling sites within a sampling class:

NOTE: Steps 1 through 5 will be completed by ERL-C. Maps that show the five random points, as discussed in step 3, will be given to each SCS sampling crew.

Step 1: Prepare a list of all mapping units and the sampling class or classes in which they occur. Most mapping units will occur only in one sampling class; complexes may occur in two or more sampling classes. For each complex, record the proportion of area occupied by each soil series in the complex (from the mapping unit description). This proportion should be the average proportion, excluding the area occupied by inclusions.

Step 2: For each watershed, obtain the watershed maps and identify the sampling classes selected for that watershed. Mapping-unit delineations for each soil series must be aggregated and identified for each sampling class.

Step 3: Transfer a grid that has a cell size of about 2 acres to a Mylar^{TM} sheet. Overlay the grid on the watershed map. Select a set of random coordinates (using a computer program) and determine if the point they represent intersects one of the sampling classes selected on that watershed. If the point does not fall within the selected sampling class, draw another pair of random coordinates. Continue this process until five random points have been identified in each sampling class. Record their order of selection from 1 through 5. Some sampling locations may not be accessible, so alternate locations must be provided.

Step 4: If the point falls on a sampling unit that is a complex, draw a random number, Y, between zero and the total percentage of the soils in the complex (e.g., a 50-30 percent complex of Tunbridge-Lyman would sum to 80, so the maximum random number is 80).

Determine the percentage of the area in the desired sampling class (e.g., Tunbridge is 50 percent). Call this number X. If X is less than Y, draw another set of coordinates. This procedure minimizes the probability that complexes will be overselected for sampling.

Step 5: For each location selected, overlay appropriate maps and note the vegetation class associated with each point as (1) coniferous, (2) deciduous, (3) mixed, (4) open dryland, or (5) open wetland.

NOTE: For comparison of coniferous, deciduous, and mixed vegetation types to Society of American Foresters (SAF) forest cover types, see Table 2.1

Within the sampling class, sample the pedons that have one or more of the soils in the sampling class and that have one or more of the vegetation classes noted above.

2.5.3 Sampling Site Locations - The procedure described above is to locate the general vicinity of the site on the watershed soil maps. This procedure is completed, and the soil maps marked with the random points are distributed, before the sampling crew leaves for the field. The point marked on the map may represent an area of 100 m^2 in the field. Within this general vicinity there may be inclusions, rock outcrops, a complex soil,

TABLE 2.1. COMPARISON OF CONIFEROUS, DECIDUOUS, AND MIXED VEGETATION TYPES TO SAF FOREST COVER TYPES

SAF Cover Type Name	Cover Type Number
Coniferous Vegetation Types	
Jack Pine	1
Balsam Fir	5
Black Spruce	12
Black Spruce - Tamarack	13
White Spruce	107
Tamarack	38
Red Spruce	32
Red Spruce - Balsam Fir	33
Red Spruce - Frasier Fir	34
Northern White Cedar	37
Red Pine	15
Eastern White Pine	21
White Pine - Hemlock	22
Eastern Hemlock	23
Deciduous Vegetation Types	
Aspen	16
Pin Cherry	17
Paper Birch	18
Sugar Maple	27
Sugar Maple - Beech - Yellow Birch	25
Sugar Maple - Basswood	26
Black Cherry - Maple	28
Hawthorn	109
Gray Birch - Red Maple	19
Beech - Sugar Maple	60
Red Maple	108
Northern Pin Oak	14
Black Ash - American Elm - Red Maple	39

(continued)

TABLE 2.1 (Continued)

SAF Cover Type Name	Cover Type Number
Mixed Vegetation Types	
Hemlock - Yellow Birch	24
Red Spruce - Yellow Birch	30
Paper Birch - Red Spruce - Balsam Fir	35
White Pine - Chestnut Oak	51
White Pine - Northern Red Oak - Red Maple	20

or other factors that make finding a soil of the specific sampling class difficult. The following procedures will be used to select the specific sampling site in the watershed.

2.5.3.1 Obtain a list of the sampling classes to be determined on that watershed. Also obtain a map that clearly shows the five predetermined random points for selection.

2.5.3.2 As best that can be determined, the sampling crew will go to the location of the first potential sampling site indicated on the map. If that location is inaccessible, go to the second potential sampling site but note the reasons in the field logbook and, if possible, on the SCS-232 field form.

2.5.3.3 If the location is accessible and the soil series at the site is in the selected sampling class and the vegetation class is appropriate, sample the pedon.

2.5.3.4 If the randomly selected site contains a soil series that is not a member of the sampling class, or if the vegetation class is not appropriate from a random-number table select a random number between 1 and 8, where 1 represents the direction north, 2 represents northeast, 3 represents east, . . . 8 represents northwest. Walk along a straight line in the direction chosen until the first occurrence of the proper combination of soil series and vegetation class is found. The maximum distance walked corresponds to a radius of 155 m around the randomly selected site. If a proper combination of soil series and vegetation class is not obtained after five tries, go the next potential site on the list. The number of trials at each site and the number of alternative sites attempted should be recorded on Form SCS-232.

These procedures provide a method for selecting a specific site and locating that site in the field.

2.6 Special Conditions

2.6.1 Inaccessible Watersheds - An attempt should be made to sample every watershed. However, some watersheds may have inaccessible areas or areas where sampling access is denied. Alternative sampling classes are selected during the random selection process as back-up sampling locations to ensure an equitable distribution of samples among sampling classes. Initial estimates of watersheds that may be remote and difficult to sample or that may be inaccessible include one in New Hampshire, one in Massachusetts, two in Connecticut/Rhode Island, three in Maine, and five in New York. Each state will formally document the reasons for excluding each watershed.

2.6.2 Inclusions - Inclusions are not representative of the soils in the sampling class and should not be sampled if the randomly selected site is located on an inclusion. The procedures described earlier accommodate this contingency. Generally, inclusions are soils associated with a sampling class other than the one being sampled. The chemical properties of the inclusion, therefore, are described when the other sampling class is sampled.

2.6.3 Agricultural Sites - The open-dryland class contains some cultivated fields. If these sites are randomly selected and access permission is obtained, the sites will be sampled. Agricultural practices, however, generally alter the chemical characteristics of the soil through fertilization, liming, and other activities.

Note samples taken from agricultural sites on the field forms. During subsequent modeling and statistical analyses, these samples may or may not be incorporated in representing watershed soil chemistry.

2.7 Paired Pedons

Paired pedon sites for sampling are selected and assigned in advance by ERL-C. These sites will be sampled in conjunction with the corresponding routine pedon. The sample code identifying the paired pedon should be treated as a routine pedon.

The location of the paired pedon is determined by the crew leader using the following criteria:

Establish sufficient distance between the two sampling locations to avoid disturbance of the paired pedon from sampling of the routine pedon.

Use the same sampling unit and vegetation class as the routine pedon.

Use the same slope position as the routine pedon.

Use the same profile description and sampling protocol as the routine pedon.

3.0 SITE AND PROFILE DESCRIPTION

3.1 Scope

Complete descriptions of the soils are essential to the soil survey and serve as a basis for soil identification, classification, correlation, mapping, and interpretation. Standards and guidelines are necessary for describing soil properties. Precisely defined standard terms are needed if different people are to record their observations so that others can understand those observations. However, the field scientist must always evaluate the adequacy of standard terms and add needed information.

The description of a body of soil in the field, whether an entire pedon or a sample within it, records the kinds of layers, their depth and thickness, and the properties of each layer. These properties include color, texture, structure, characteristics of failure and disruption, roots and animals (and their traces), reaction, salts, and boundaries between layers. Some properties that apply to the entire sampling unit are also measured and recorded. Generally, external features are observed throughout the extent of the polypedon; internal features are observed from study of a pedon that is judged to be representative of the polypedon.

For a soil description to be of greatest value, the part of the landscape that the pedon represents should be known and recorded. Descriptions of pedons that represent an extensive mappable area are generally more useful than are descriptions of pedons that represent the border of an area or a small inclusion. Consideration is given to external and internal features of the soil, related features such as vegetation and climate, and the setting--the position of the particular soil in relation to other soils and to the landscape as a whole.

Pedons used for detailed study of a soil are selected tentatively at first. Areas that previous studies have shown to contain the kind of soil to be described and sampled are most commonly chosen. The pedon is usually selected on the basis of external evidence. Depending on the purpose of the study, the selected pedon may be one that has properties either near the middle of the range of the taxon or near the limits of the range. After a sampling site is tentatively located, it is probed with an auger, spade, or sampling tube to verify that the soil at the site does have the diagnostic features of the soil and that its properties at the site represent the desired segment of the soil's range.

A pit that exposes at least one clean, vertical face (approximately 1 m across) to an appropriate depth is convenient for studying most soils in detail. Horizontal variations in the pedon, as well as features too large or too widely spaced to be seen otherwise, can be observed. The sides of the pit are cleaned of all loose material disturbed by digging. The exposed vertical faces are then examined starting at the top and working downward, to identify significant differences in any property that would distinguish between adjacent layers. Boundaries between layers are marked on the face of the pit, and the layers are identified and described.

Photographs can be taken after the layers have been identified but before the vertical section has been disturbed for description. If point counts are to be made for estimation of volume of stones or other features, the counts are made before the layers are disturbed. If samples are to be taken to the laboratory for analyses or other studies, they are collected after the soil has been described.

Horizontal relationships between soil features can be observed in a cross section of each exposed layer by removing the soil above it. Each horizontal section must be large enough to expose any structural units. A great deal more about a layer is apparent when it is viewed from above, in horizontal section, as well as in vertical section. Structural units that are otherwise not obvious, as well as the third dimension of many other features, can be seen and recorded. Patterns of color within structural units, variations of particle size from the outside to the inside of structural units, the pattern in which roots penetrate structural units, and similar features are often seen in horizontal section more clearly than in a vertical exposure.

3.2 Field Properties

The following parameters will be determined in the field by established SCS methods and protocols[1,2,4]:

- Horizon type
- Horizon depth
- Color
- Texture
- Structure
- Consistence
- Boundary type
- USDA/SCS soil taxonomic designation
- Surface vegetation type and abundance
- Parent material
- Physiography

- Relief
- Slope
- Aspect
- Permeability
- Erosion class
- Root distribution
- Drainage class
- Depth to bedrock
- Bedrock exposure
- Volume percent coarse fragments by visual estimation

    20 to 75 mm

    75 to 250 mm

    >250 mm

- Diagnostic features

- Mottle type and abundance

The field crew will use Form SCS-SOI-232 for field description which is coded for easy input onto a computerized data file. The protocol for horizon description is discussed in detail in the SCS Soil Survey Manual[2], the SCS National Soils Handbook[1], and Principles and Procedures for Using Soil Survey Laboratory Data[3].

3.3 Profile Excavation

The exposed face of the pedon must be wide enough to permit pedon description, the collection of bulk-density clods, and the collection of 5.5 kg or more of sample from each of the significant horizons. The pedon face should be photographed (Section 3.4) before destructive sampling begins.

3.4 Photographs of Profile and Site

Photographic documentation of the sampling phase will be useful for later reference and future discussions concerning specific site considerations, and will complement field descriptions. Field crews will provide their own single-lens reflex, 35-mm cameras or equivalent and will obtain film locally. Ektachrome, ASA 400 slide film is recommended, but field crews should determine film speed suitability based on their knowledge of the site. If flash attachments and tripods are available, they should be included in the sampling equipment. For film-quality consistency, all slides should be developed using prepaid Kodak mailers.

Photographic documentation requires that a precise logbook be kept to identify corresponding slides. The indexing system can be developed by the field crew, but must be based on the sample code from NADSS Label A to identify the site. The system must be fully explained in the logbook. Once the slides have been developed, they should be labeled on the slide mounts with the sample code and any other information the field crew deems necessary. Slides will be stored in 3-ring binders in slide files and will be submitted with the logbook to ERL-C at the conclusion of the sampling phase of the survey. Histosols should be photographed by sequential placement of the augered horizons on the surface.

The pedon face, tree canopy, understory vegetation, and representative landscape or landform will be photographed for each site sampled. Scale should be provided by including a meter stick, rule, or other suitable item in the photograph. Pedon face identification can be positively made by including NADSS Label A or an index card displaying Label A information in the photograph.

SCS protocols for field photography are outlined in the SCS National Soil Survey Manual[2], Chapter 9.

3.5 Important Points Concerning Horizon Descriptions

The sample site should be free of road dust and chemical contamination. State all known spraying of pesticides and herbicides.

Soils will be sampled only from freshly dug pits large enough (1 m x 1 m) to allow sampling of all major horizons to a depth of 1.5 m or to bedrock.

Samples will be taken from continuous horizons >3 cm thick, including the C horizon if present. Discontinuous horizons will be sampled when considered significant by the crew leader.

Clods will be collected for all horizons sampled, except the O_i horizon. The bulk density procedure is detailed in Section 7.0.

All obvious horizons in a pedon are to be sampled, although a maximum of six horizons had been previously specified as a limit for cost estimates and planning purposes. It is the decision of the field soil scientist whether or not a horizon is significant enough, for DDRP purposes, to be sampled and described. Therefore, if the field soil scientist believes there are eight significant horizons, he should sample all eight.

Pedons can not be dug in wetlands. The recommended procedure for obtaining a 5.5-kg sample is to use a peat-sampling corer.

Sample pits will be accurately located on the soil survey maps, and the pit dimensions and the azimuth perpendicular to the pit face will be recorded. The location of the pit in the field should be flagged or identified so that it can be revisited, except in areas where this is not possible due to landowner restrictions. One horizon per day will be sampled twice by each field crew. This will be the field duplicate (FD). The choice of which horizon to duplicate is at the discretion of the field crew. The procedure for obtaining this duplicate sample is to alternate when placing trowel or shovelfuls of sample into each sample bag. The horizon that is chosen for a field duplicate should be alternated each day so that a complete range of field duplicates by horizon is achieved.

3.6 Field Data Form - SCS-232

All field data should be recorded on Form SCS-SOI-232, which is reproduced along with a modified legend in Appendix A. The SCS is responsible for making sure that completed copies of these forms are sent weekly to the following groups:

One copy to the preassigned soil preparation laboratory for each crew.

One copy to the EPA Environmental Monitoring Systems Laboratory-Las Vegas (EMSL-LV) to:

Lockheed Engineering and
Management Services Company, Inc.
1050 E. Flamingo, Suite 120
Las Vegas, Nevada 89109

One copy to Oak Ridge National Laboratory (ORNL) to:

Oak Ridge National Laboratory
P.O. Box X
Building 1505, Room 343
Oak Ridge, Tennessee 37831

and one copy to the EPA ERL-C to:

Environmental Research Laboratory-Corvallis
200 S.W. 35th Street
Corvallis, Oregon 97333

NOTE: The following changes and additions from the normal procedure should be made to complete Form SCS-232.

Page 1 of 4

Under "Sample Number," "unit" is synonymous with "pedon."

Under "Date" add the day as: __/__/__ Month Year Day

Under "Describers Name" add the Crew ID in the upper right hand corner.

Under "Location Description and Free Form Site Notes" the first six digits of line 1 should be the site ID (Lake ID), the seventh digit is a dash, the eighth digit is the random number point (1 to 5), the ninth digit is a dash and digits 10 through 12 are the sampling class, digit 13 is a dash, digits 14 through 16 are the azimuth perpendicular to the described pit face, and digit 17 is a degree symbol "°".

Under "Vegetation" describe the three major species by decreasing basal area. Clearcut should be noted as "CC." Describe dominant vegetation types prior to clearcut in the free form site notes.

The following soil description parameters need not be completed by field crews, but may be if information is accessible: PRECIP, TEMPERATURES °C, WEATHER STATION NUMBER, ER.

Page 2 of 4

Dry color should be determined when needed for classification.

"VOL (LAT/TOT)" need not be completed but may be if information is accessible.

Page 3 of 4

Mottles should be described as indicated in Chapter 4 of the National Soils Survey Handbook[1].

"EFFERVESCENCE" will be determined at the preparation laboratory and need not be completed here.

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The three divisions under "ROCK FRAGMENTS" correspond to the three volume particle size estimates:

line 1 = 2 to 75mm
line 2 = 75 to 250mm
line 3 = >250 mm

Legend

Under "SITE DESCRIPTION CODES" for page 1 add "AA" for a local site description.

4.0 SAMPLING PROCEDURES

4.1 Scope

The objective of the field sampling phase of the DDRP is to characterize the soil and watershed characteristics across the regions of concern, the northeastern United States, and the southwestern portion of the Blue Ridge Province.

Field sampling includes the collection of a 5.5-kg field sample that will yield a minimum of 2 kg of air-dried material of particle sizes <2 mm. This requires 5.5 kg of mineral soil, or as much soil possible to fill the presupplied 46 cm x 53 cm sample bags, and twice this volume for organic soils. In addition, bulk-density clods will be sampled for laboratory determination of field bulk density.

4.2 Sampling the Pedon

4.2.1 Field Sampling Protocols - Field sampling protocols are based on the standard methods routinely used by SCS. The following procedural steps were developed by the National Soil Survey Laboratory, Lincoln, Nebraska, and are detailed in a publication titled *Principles and Procedures for Using Soil Survey Laboratory Data*[3]. An edited version of these procedures is reproduced here. The protocol for collecting bulk-density samples is specified in Chapter 7.0 of this manual.

4.2.2 Sampling Party Responsibilities - The sampling party has responsibility to obtain samples representative of the pedons selected for characterization. Although some sampling protocol has been specified, field-crew decisions are necessary on how deep to sample, horizon delineation, thickness of horizon (or interval) sampled, what material should be excluded from the sample, and the usefulness of compositing samples. The sampling party ensures that site and pedon descriptions are adequate.

4.2.3 Pedons for Characterization - Pedons for characterization studies should be sampled to a depth of 1.5 m where possible. In cases where the lower depths of the profile appear homogenous and the C horizon material is particularly difficult to penetrate in (e.g., dense basal till), it may not be feasible to dig the pit to 1.5 m. However, it is still possible that a dense basal till will show a variable pH from the upper to the lower sections of the C horizon. If this were true, a sample would be desirable even if the material is hard to dig. These types of decisions are judgments to be made to the best of the ability of the sampling-crew leader and should be documented in the field sampling notebook. The sampling party needs to be alert to taxonomic questions that may arise and sample appropriately to resolve the questions (i.e., base saturation for Alfisol versus Ultisol may require subsampling at a specific depth). Appropriate sampling increments depend on the kind of material and the proximity of the horizon to the soil surface. Horizons in the upper 1 m would usually be split for sampling if they are more than 30 cm thick, excluding organic horizons. Uniform horizons below 1 m are usually split for sampling if they are more than 75 cm thick. The sampling party must exercise good judgment in this decision process. The ideal sample contains each soil material within the horizon in proportion to its occurrence in the pedon. The sampler attempts to approximate the ideal by carefully sampling a selected section of the horizon. The sample is usually taken along a pit face from horizon boundary to horizon boundary and between arbitrary lateral limits.

4.2.4 Lateral Limits - Lateral limits encompass short-range variability observed at the site. If a recurring pattern (i.e., mottles, durinodes, nodules, plinthite) is discerned, extend the lateral limits to four or five cycles of the pattern. If this produces too much material, the sample is mixed, quartered, and subsampled. At some point, the repeat cycles become too large or soil properties change sufficiently that lateral extension is impractical or undesirable. An example is the gilgai pattern in Vertisols. Proper characterization may warrant the sampling of two sets of horizons or pedons.

4.2.5 Stratified Horizons - If a horizon is stratified or otherwise contains contrasting materials, each material should be carefully described. Some contrasting materials can be sampled independently, but in many cases the materials are intertwined to the point that practicality dicates they be sampled together. Each material should be described and the proportions should be noted, however. A decision on what, if any, materials should be excluded from the sample is an integral part of collecting a representative sample. The sampling party may decide to include soil material in cicada casts and nodules as part of the sample, but to exclude material from a badger tunnel.

Coarse fragments (>20 mm) will be excluded from all samples sent to the laboratory except for bulk-density clods.

4.2.6 Composite Samples - One sampling technique designed and used here to average lateral variability is to sample three or four relatively small segments (20 to 30 cm wide) of the same pedon at several points around the pit. The samples are composited, are mixed, and a representative sample is sent to the laboratory for analysis.

4.2.7 Filling Sample Bag - Approximately 5.5 kg or more of soil less than 20 mm in diameter should be placed in each plastic sample bag. However, the amount of soil obtained for chemical analysis is highly dependent on the amount of coarse fragments contained in each horizon.

For example, if the horizon is determined to contain 50 percent coarse fragments by a visual estimate, the corresponding weight estimate for coarse fragments is 65 percent (Table 4.1). This estimate indicates that a 5.5-kg sample will contain 35 percent of material <2 mm or only

1.8 kg of sample. Field sampling protocols specify that a minimum of 2 kg of soil of particle size <2 mm is necessary for the chemical and physical analyses specified. Care must be taken to ensure that field samples will yield the minimum 2 kg of soil in the <2 mm particle-size class. Table 4.1 illustrates that a 5-kg sample from horizons containing coarse fragments greater than 60 percent by weight or 45 percent by volume will not be sufficient to obtain a minimum 2-kg sample. Minimum sample weights for horizons with coarse fragments and weights in this catagory are provided in Table 4.1.

NOTE: This table is included as a guide and probably will not be most useful in the field, but the concept explained is important.

The general rule to follow is that the minimum amount of field sample is 5.5 kg of the ≤20-mm particle-size fraction. If the estimated 2- to 20-mm size class exceeds 45 percent by the volume estimate, then two 5.5-kg samples or two full sample bags of mineral soil are necessary. Two full bags of organic horizon material are requested in every case possible. Plastic sample bags should be pre-labeled with NADSS Label A. Attach the label to the center of the bag, not near the top of the bag. Double check that all designations are correct, complete, and legible. Large, easily removed nonmineral material should not be included in the sample. Limit handling of the soil sample to avoid contamination.

TABLE 4.1. VISUAL ESTIMATE OF PERCENT VOLUME OF ROCK FRAGMENTS GREATER THAN 75 mm CORRELATED TO PERCENT WEIGHT

% Volume	% Weight	Weight of <20 mm particles in a 5-kg sample	Sample weight required to obtain a minimum 2-kg sample
0	0	5.00	5.0
3	5	4.75	5.0
7	10	4.50	5.0
10	15	4.25	5.0
13	20	4.00	5.0
16	25	3.75	5.0
20	30	3.50	5.0
23	35	3.25	5.0
27	40	3.00	5.0
31	45	2.75	5.0
35	50	2.50	5.0
40	55	2.25	5.0
45	60	2.00	5.0
50	65	1.75	5.7
56	70	1.50	6.6
62	75	1.25	8.0
68	80	1.00	10.0
74	85	0.75	13.0
80	90	0.50	20.0

In wet soils, such as Histosols, excess water should be drained before sealing the sample bag.

The top of the plastic sample bags should be folded down in 2.5-cm sections. The folded sections should then be stapled or tied with twist-ties to seal.

The plastic bags should then be placed within pre-labeled canvas bags. Label the canvas bag below the center with indelible ink or use pre-supplied label stamps. Record exactly the same information contained on NADSS Label A. Seal the canvas bag by tying or stapling. Place the samples in coolers with Blue Ice^{TM} as soon as possible after field sampling. Transport samples to the preparation laboratory as soon as possible.

4.2.8 NADSS Label A (Figure 4.1) - The date sampled is entered in the format DD MMM YY. For example, March 14, 1985, will be 1 4 M A R 8 5. The crew ID will consist of four digits: the first two are alphabetic, representing the state; the second two are the number assigned to each crew for the state, for example, NY01. The site ID consists of six digits and appears on the assigned watershed map as:

$\overline{1}$	$\overline{2}$	$\overline{3}$	$\overline{4}$ $\overline{5}$ $\overline{6}$
Region	Subregion	Alkalinity Class	Watershed ID

The sample code represents the SCS (FIPS) soil ID code and the sample type. The first three digits of the sample code represent the type of sample (R11 = routine sample, one bag, one sample; R23 = routine sample, 2nd of 3 bags; R33 = routine sample, 3rd of 3 bags; Field Duplicate = FD0, [FD1, FD2 are used for compound bags of field duplicates] etc.), digits 4 to 5 are the SCS state code, 6 to 8 are the SCS county code, digit 9 is a dash, digits 10 to 11 are the county pedon number and

NADSS Label A

Date Sampled: D D M M Y Y

Crew ID: _ _ _ _

Site ID: _ _ _ _ _ _

Sample Code: _ _ _ _ _ _ _ _ _ _ _ _ _

Horizon: _ _ _ _ (Depth)

Set ID: _ _ _ _ _

Figure 4.1. NADSS Label A.

digits 12 to 13 are the horizon number. Upper and lower split horizons will be identified by the depth designations (written after the horizon designation). A "U" or an "L" can also be written after the horizon depth to help to differentiate these samples for the preparation laboratories. The Set ID is a four-digit number beginning with 0. The field sampling crews are assigned the following ideal set of 100 Set ID numbers for sampling in the Northeast:

100-199	ME02	700-799	MA01
200-299	ME03	800-899	MA02
300-399	NH01	900-999	CT01
400-499	NY01	1000-1099	PA01
500-599	NY02	1100-1199	VT01

The field sample will be passed through a 75-mm sieve. All coarse fragments remaining on the sieve can be subdivided manually into two size classes; 75 to 250 mm and $\geq$250 mm. An estimate will be made of the volume percent of material in these classes. A volume estimate of the percent coarse fragments for the 20- to 75-mm fraction will be made as well. This information will be entered on SCS Form 232 under the Rock Fragments category, Size (SZ, 1 = 20 to 75 mm, 2 = 75 to 250 mm, and 3 = $\geq$250 mm). The preparation laboratory will determine the percent coarse fragments in the 2- to 20-mm fraction. The sieved soil <20 mm should be used as the soil sample and should be placed in the sample bag according to procedures in Section 4.2.7.

4.3 Delivery

The soil samples should be delivered to the pre-assigned soil preparation laboratory. The following preparation laboratory assignments are for the Northeast sampling crews. Preparation laboratories for the southeastern sampling crews will be assigned at a later date.

Field Crew	Preparation Laboratory
ME	University of Maine
NH, VT, MA	University of Massachusetts
CT, RI	University of Connecticut
NY, PA	Cornell University

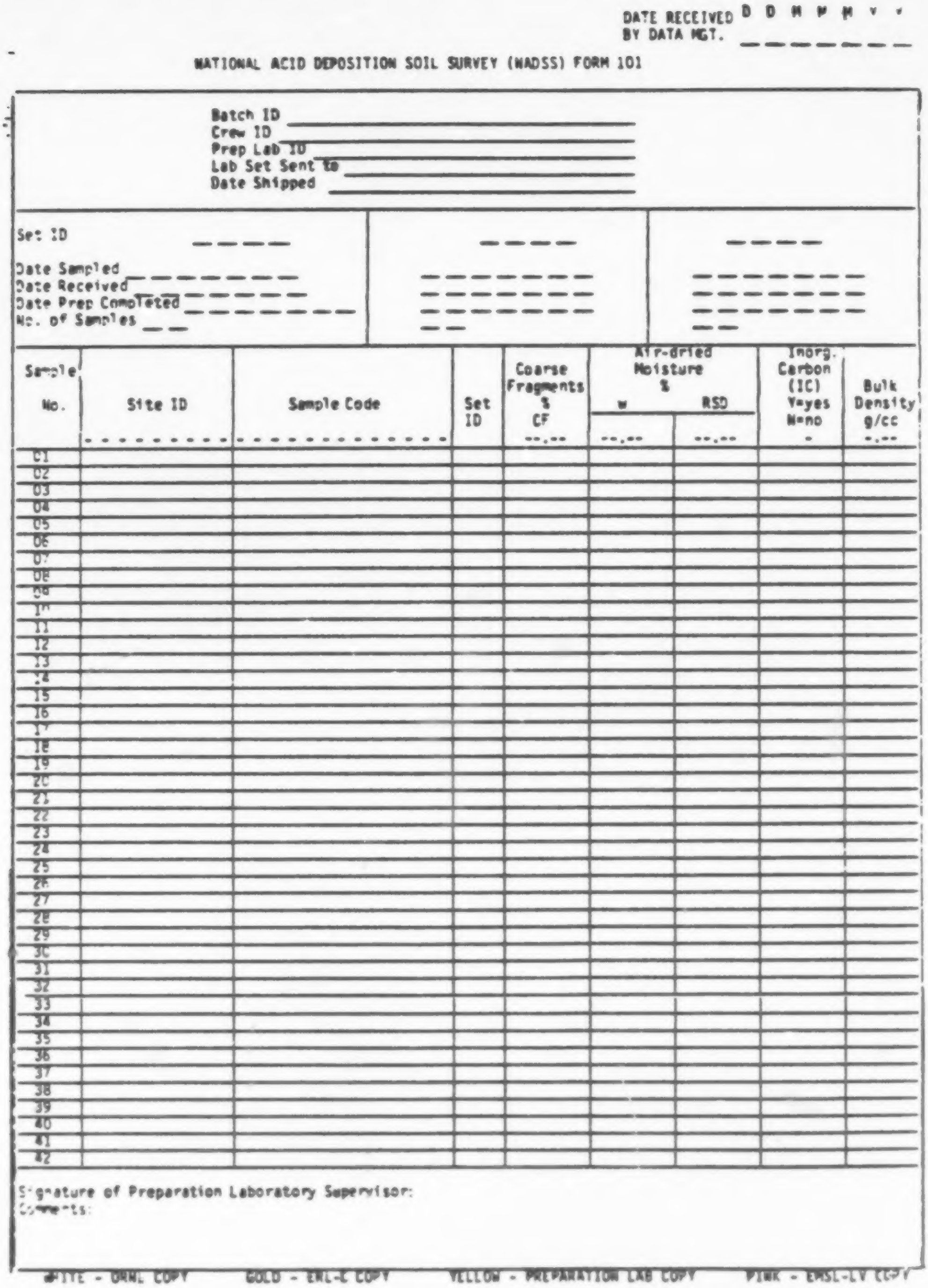
Samples will be kept as cold as possible in the field by storage in coolers with Blue Ice^{TM} gel packs until delivery to the preparation laboratory. Temperature checks in the cooler should be made routinely to keep a 40°C ambient air temperature. These readings should be recorded in the field logbook. Due to the location of some watersheds, some samples may not be delivered to the preparation laboratory until three to four days after they are sampled. Each field sampling crew will deliver field samples as soon as possible after collection. If major problems occur, notice must be given as soon as possible to the QA Officer Every effort should be made to get the field samples to the preparation laboratory as soon as possible.

Great care should be taken not to drop or puncture sample bags in transport to the preparation laboratory.

5.0 SOIL PREPARATION LABORATORY

5.1 Scope

The samples will be received by the preparation laboratory supervisor. The supervisor will check the samples for spillage or other problems and to be certain that each sample has an accompanying NADSS Label A (Figure 4.1). Field samples and all QC samples will be logged in on NADSS Form 101 (Figure 5.1). The QC samples will be randomly assigned in the batch by the preparation laboratory. One set of samples will be defined as the total number of samples taken in one day by one crew. Each set will include one field duplicate, because one horizon per day is to be sampled twice as a field duplicate. Two pre-assigned audit samples will be randomly inserted into each batch. In addition, one sample per batch will be randomly selected, will be divided into two samples, and will be tracked as the preparation laboratory duplicate (PLD). One batch including routine field samples, field duplicates, a preparation laboratory duplicate, and 2 audit samples will contain a maximum of 42 samples. Therefore, the number of sets combined to make one analytical batch depends on the number of samples in each set. The total number of samples in the combined sets should not exceed 39.



DATE RECEIVED D D M M M Y Y
BY DATA MGT. _ _ _ _ _ _ _

NATIONAL ACID DEPOSITION SOIL SURVEY (NADSS) FORM 101

Batch ID ____________________
Crew ID ____________________
Prep Lab ID ____________________
Lab Set Sent to ____________________
Date Shipped ____________________

Set ID _ _ _ _	_ _ _ _	_ _ _ _
Date Sampled _ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _
Date Received _ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _
Date Prep Completed _ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _
No. of Samples _ _	_ _	_ _

Sample No.	Site ID	Sample Code	Set ID	Coarse Fragments % CF	Air-dried Moisture % w	RSD	Inorg. Carbon (IC) Y=yes N=no	Bulk Density g/cc
	- - - - - - - - -	- - - - - - - - - - - - - -		--.--	--.--	--.--	-	-.--
01								
02								
03								
04								
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42								

Signature of Preparation Laboratory Supervisor:
Comments:

WHITE - ORNL COPY GOLD - ERL-C COPY YELLOW - PREPARATION LAB COPY PINK - EMSL-LV COPY

Figure 5.1. National Acid Deposition Soil Survey (NADSS) Form 101.

5.2 Sample Storage

The samples will be sealed and will be stored at 4°C at all times when not involved in processing. This procedure will greatly reduce microbial decomposition of organic matter without alteration of the crystalline structures. If the samples cannot be dried immediately at the preparation laboratory, they should be placed in storage until processing.

5.3 Sample Preparation

After the samples are received, sample numbers are assigned on NADSS Form 101. The samples should be air-dried and sieved (<2 mm) (see Section 5.3.1). Care must be taken to be certain that the soils are not separated from their labels during the air-drying process. The percentage of coarse fragments (>2 mm) must be weighed as specified in Section 5.3.2 and the percent coarse fragments reported on NADSS Form 101. The coarse-fragment fraction should be labeled and set aside. If the qualitative test for inorganic carbon is positive, the analysis for total inorganic carbon must be performed on this sample, and the 2- to 20-mm fraction must be crushed and shipped to the analytical laboratory. The results of the determination of effervescence are recorded on NADSS Form 101.

5.3.1 Sample Drying and Mixing

5.3.1.1 The soil is laid out on a tray and allowed to air-dry at room temperature until constant weight is achieved (30 to 35°C is ideal). Constant weight is defined as that time when a subsample does not change by more than 2.5 percent moisture content on two consecutive days. Constant weight must be determined before the sieving process is started. The drying period could range from two days to seven or more days, depending on organic matter content and particle size of the sample.

5.3.1.2 After the soil is air-dried, place the complete sample in the original sample bags and store them at 4°C until further preparation laboratory analysis is performed.

5.3.1.3 After the soil is air-dried, place the complete sample minus the calibration sample in the original sample bags and store them at 4°C until preparation laboratory analysis.

5.3.2 Coarse Fragment Determination

5.3.2.1 The fragment size class that will be separated during this procedure is the class that is small enough to pass through a 20-mm sieve. Coarse fragments larger than 20 mm will be determined in the field.

5.3.2.2 The total sample should be weighed and quantitatively passed through a clean, dry, square-holed, 2-mm sieve to segregate coarse fragments (2 mm to 20 mm) from the soil. The material larger than 2 mm should be saved until the test for inorganic carbon is complete. The soil that passed through the sieve (0 to 2 mm) should be placed in a sealed container if further processing will not occur at this time.

5.3.2.3 The amount of soil that did not pass through the sieve should be weighed and should be divided by the initial amount and should be multiplied by 100. This percentage is then recorded as percent coarse fragments (%CF). The coarse fragments (2 to 20 mm) must be saved until the qualitative test for inorganic carbon has been completed.

5.3.3 Soil Mixing - After the soil has passed through a 2-mm sieve and %CF is determined, quantitatively load the soil into the Jones type 3/8-inch riffle splitter. The soil should be passed through the riffle splitter at least seven times. Before reloading the splitter each time, level the soil on the tray to ensure random particle addition. It is best to remove the 1-kg subsample for the analytical laboratory at this time. If the 1-kg subsample is to be removed later, the entire sample must again be passed through the riffle splitter before a well-mixed subsample can be removed. After completion of the soil preparation procedures, the soils should be placed into a new inner plastic liner supplied by EPA-LV. Complete NADSS Label B (Figure 5.2) and place it on the exterior of the inner bag that is to be sent to the analytical lab.

Batch ID: _ _ _ _ _ _

Sample No.: _ _

Figure 5.2. NADSS Label B.

Remove NADSS Label A from the original field bag and tape it into a preparation laboratory notebook, grouped in order by set number and batch number. Record the date either on the label or below it. Initial the label by writing partially on the label and partially on the page. This procedure will help to replace labels that may become unattached. The air-dried soil in the inner bag should be sealed with a plastic-coated wire twist.

At this point, the exterior canvas bag will have the field coding written on it and the inner bag will show the batch number and sample number.

The field coding on the outer plastic bag should be crossed out so it is not legible, and the batch number and sample number should be written on the exterior with indelible ink. The soil should be packed tightly in the boxes supplied by EPA-LV.

After all subsamples have been removed for shipment to the analytical laboratories, the remaining sample should be placed in a clean plastic bag and should be stored at 4°C. The samples should be clearly and permanently labeled with NADSS Label B and should be stored in such a manner that they are easily retrievable if necessary.

5.3.4 Qualitative Test for Inorganic Carbon

5.3.4.1 Carbonates are used frequently as criteria to differentiate soil series. A qualitiative test for carbonates will be performed on the $\leq$2-mm size class. If the test for effervescence is positive, the coarse-fragment size class (2 to 20 mm) will be crushed and sent to the analytical laboratory for quantitative total inorganic carbon analysis. For the following procedures, the word "soil" is defined as that material which has been air-dried and passed through a 2-mm sieve.

5.3.4.2 Place 1 g of soil in a porcelain spot plate. Saturate the soil with DI water and stir with a glass rod to remove entrapped air. Place plate under a binocular microscope.

5.3.4.3 Add 4 N HCl by dropwise addition and observe through microscope for effervescence.

5.3.4.4 Repeat this procedure with another 1 g of soil from the sample.

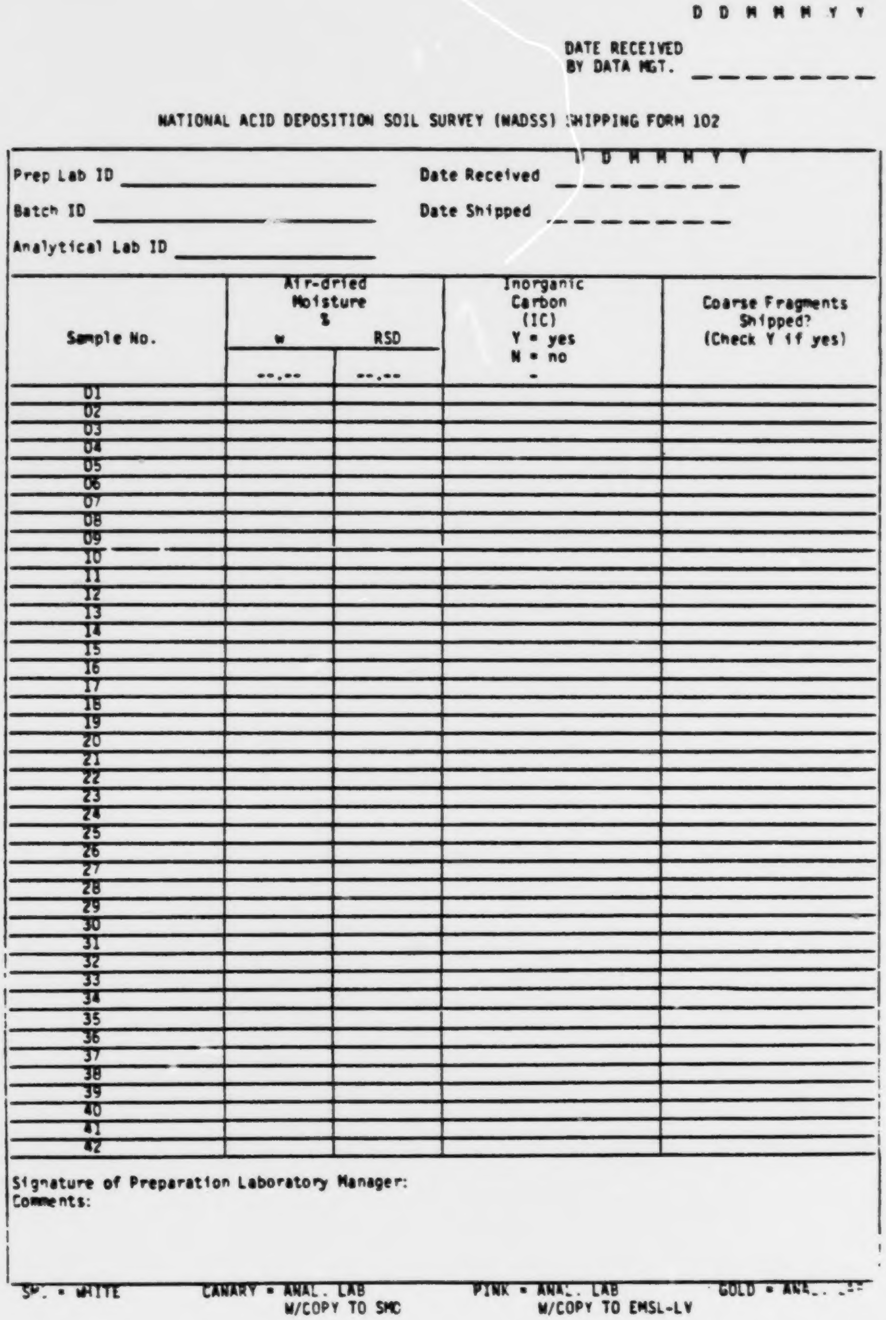
5.3.4.5 Record in laboratory notebook for each subsample whether effervescence was or was not noted.

5.3.4.6 If effervescence was noted either time, inorganic carbon must be determined for this sample.

5.3.4.7 If effervescence was observed, the coarse-fragment fraction from this soil sample should be crushed to pass an 80-mesh sieve. A 100-gram subsample should be prepared using a riffle splitter, and should be shipped separately (with the soil sample) for inorganic carbon determination. The subsample should be packaged in a plastic bag and labeled with NADSS Label B. Coarse-fragment subsamples do not require storage at 4°C until shipment to the analytical laboratory.

5.4 Shipment of Subsample to Analytical Laboratories

5.4.1 Shipping Method - Subsample will be shipped to the analytical laboratories by batch. Each box shipped must contain copies of NADSS Shipping Form 102 (Figure 5.3). The results of the bulk density determination and percent coarse fragment determinations must also appear on Form 102. If Form 102 indicates a positive inorganic carbon test, the coarse-fragment sample must be shipped to the analytical laboratory for total inorganic carbon analysis. As indicated on the bottom of NADSS Form 101, the canary, pink, and gold copies should be enclosed with each



D D M M M Y Y

DATE RECEIVED
BY DATA MGT. _ _ _ _ _ _ _

NATIONAL ACID DEPOSITION SOIL SURVEY (NADSS) SHIPPING FORM 102

Prep Lab ID ____________________ Date Received _ _ _ _ _ _ _ (D D M M M Y Y)

Batch ID ____________________ Date Shipped _ _ _ _ _ _ _

Analytical Lab ID ____________________

Sample No.	Air-dried Moisture % w	RSD	Inorganic Carbon (IC) Y = yes N = no	Coarse Fragments Shipped? (Check Y if yes)
	--.--	--.--	-	
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
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42				

Signature of Preparation Laboratory Manager:
Comments:

SP. = WHITE CANARY = ANAL. LAB W/COPY TO SMC PINK = ANAL. LAB W/COPY TO EMSL-LV GOLD = ANA[illegible]

Figure 5.3. National Acid Deposition Soil Survey (NADSS) Shipping Form 102.

sample box. The white copy should be sent to the Sample Management Office (SMO) after a photocopy is made to keep at the preparation laboratory. The address for shipment to SMO is:

National Acid Deposition Soil Survey
Sample Management Office
P. O. Box 818
Alexandria, Virginia 22313

The shipping carrier to be used and specific shipping protocols required to ship samples to the analytical laboratory will be supplied to the preparation laboratory by the QA Manager.

5.4.2 NADSS Form 101 - NADSS Form 101 is used to combine field sets into an analytical set. A maximum of six sets should be combined to achieve a maximum of 39 routine and field duplicate samples. In addition, there will always be one preparation laboratory duplicate (PLD) and two audit samples per batch for a combined maximum number of 42 samples. If four to six sets are used for one batch, the second section of Form 101 should be modified to fit, ignoring the predrawn lines and utilizing space as necessary. Air-dried moisture (or column "w") should be the final mositure content used to verify air-dryness, reported to two decimal places.

NADSS Form 101 should be completed in black ink and should not contain any mistakes, crossouts, or white out. The form should be mailed within 24 hours after the batch has been shipped to the analytical laboratory. The white copy should be sent to ORNL at the following address:

Oak Ridge National Laboratory (ORNL)
P.O. Box X
Building 1505, Room 343
Oak Ridge, Tennessee 37831

The gold copy should be sent to the EPA ERL-C, in care of:

Environmental Research Laboratory, Corvallis
200 S.W. 35th Street
Corvallis, Oregon 97333

the pink copy should be sent to the EPA EMSL-LV, in care of:

Lockheed Engineering and Management
Services Company, Inc.
1050 E. Flamingo, Suite 120
Las Vegas, Nevada 89109

5.5 Sample Receipt by the Analytical Laboratory from the Preparation Laboratory

The analytical laboratory should immediately compare the samples and the data on Form 102. Record should be made as to when the samples were received, their condition upon receipt. All missing samples should be noted. This information should be recorded on Form 102 and should be initialed by the recipient.

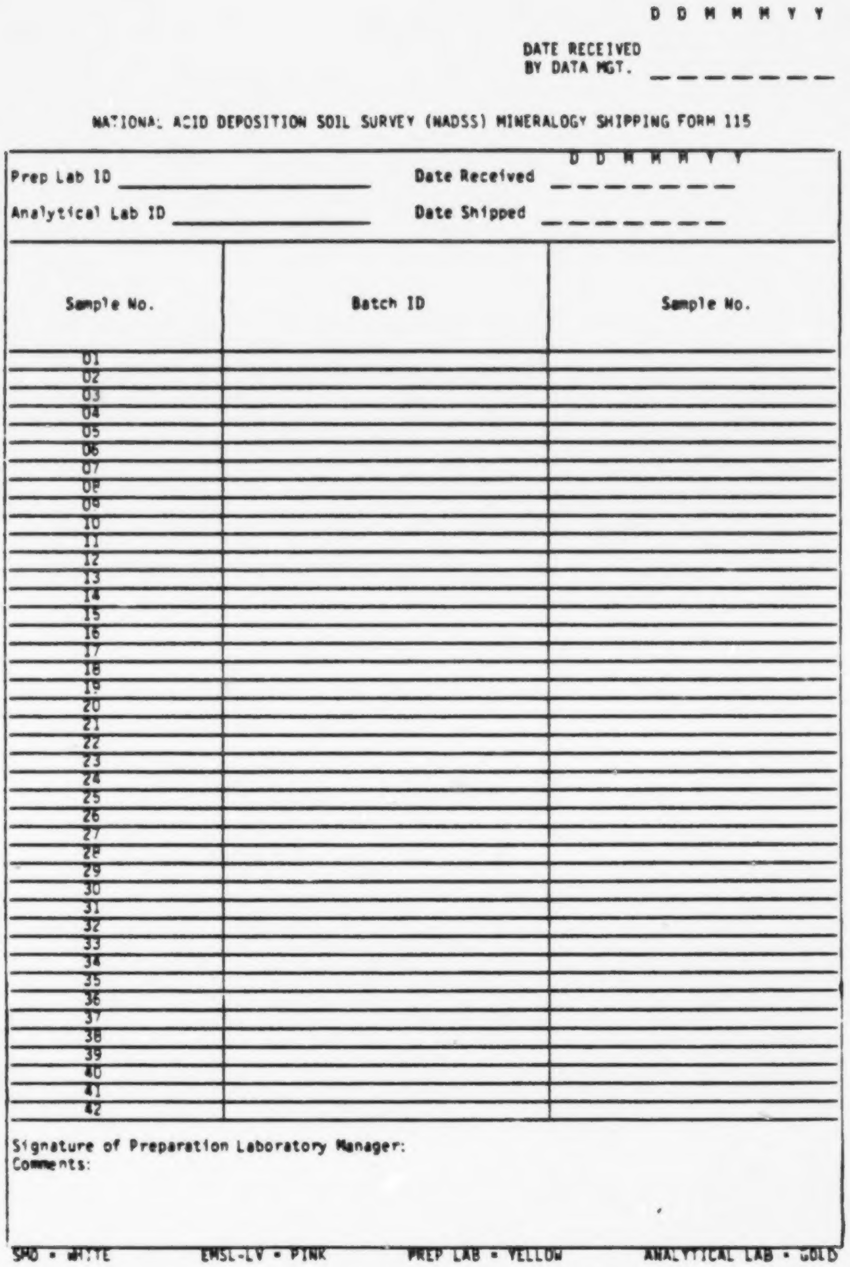
If NADSS Form 102 is incomplete, immediately notify SMO at (703) 557-2490. The gold NADSS Form 102 should be kept at the analytical laboratory. The canary NADSS Form 102 should be sent to SMO at the address indicated in Section 5.4 and the pink copy should be mailed to EMSL-LV at the following address:

Lockheed Engineering and Management
Services Company, Inc.
1050 E. Flamingo, Suite 120
Las Vegas, Nevada 89109

The recipient should check to be sure that all samples for inorganic carbon analysis have been included.

5.6 Shipment of Mineralogical Samples

Horizons to be subsampled for mineralogical analysis will be designated by the QA Manager. Approximately 10 percent of the pedons sampled will require this analysis. Subsamples (100 g each) for mineralogical analysis will be placed in 250-mL polyethylene bottles supplied by EMSL-LV. NADSS Label B (Figure 5.2) will be placed on these bottles and shipping Form 115 (Figure 5.4) will be included in each box shipped. Sample receipt protocol by the mineralogical laboratory is the same as that specified in Section 5.4 for analytical samples.



D D M M M Y Y

DATE RECEIVED
BY DATA MGT. _ _ _ _ _ _ _

NATIONAL ACID DEPOSITION SOIL SURVEY (NADSS) MINERALOGY SHIPPING FORM 115

Prep Lab ID ________________ Date Received _ _ _ _ _ _ _ (D D M M M Y Y)

Analytical Lab ID ________________ Date Shipped _ _ _ _ _ _ _

Sample No.	Batch ID	Sample No.
01		
02		
03		
04		
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42		

Signature of Preparation Laboratory Manager:
Comments:

SMO • WHITE EMSL-LV • PINK PREP LAB • YELLOW ANALYTICAL LAB • GOLD

Figure 5.4 National Acid Deposition Soil Survey (NADSS) Form 115

6.0 SUMMARY OF PHYSICAL AND CHEMICAL PARAMETERS AND METHODS

6.1 Physical Parameters

6.1.1 Particle Size - Soil-texture analysis is routinely determined for soil characterization and classification purposes. The standard pipet method is used. Particles greater than 20 mm will be determined by field sieving and weighing, coarse fragments (2 to 20 mm) will be determined at the soil preparation laboratory, and soil less than 2 mm will be determined at the analytical laboratory. This analysis will be performed on all mineral horizon samples, including the additional samples from each impervious layer less than 3 cm thick.

6.1.2 Mineralogy - Clay minerals are identified by x-ray diffraction, whereas light and heavy minerals of the fine-sand fraction are identified by optical mineralogy. Mineralogical identification is necessary to (1) help characterize the soil, (2) provide an indication of weathering rates, and (3) yield information about minerals weathered from the parent material. This analysis will be performed only on samples selected by ERL-C.

6.1.3 Specific Surface Area - Specific surface is measured because this is highly correlated with anion adsorption/desorption, cation exchange capacity, and the type of clay mineral. The method specified is saturation with ethylene glycol monomethyl ether. This analysis will be performed on all mineral horizon samples.

6.2 Chemical Parameters

6.2.1. pH - pH is a measurement of free hydrogen ion activity. pH measurements are determined in three different soil extracts. The extracts are deionized (DI) water 0.01 M $CaCl_2$, and 0.002 M $CaCl_2$ in a 1:2 ratio in a mineral soil and a 1:5 ratio for organic horizon samples. These analyses will be performed on all samples.

6.2.2 Total Carbon and Total Nitrogen - Total carbon and total nitrogen are critical parameters due to their close relationship with microbial decomposition of soil organic matter. The method specified is oxidation followed by thermal conductivity detection using an automated CHN analyzer. These analyses will be performed on all samples.

6.2.3 Inorganic Carbon - Quantification of inorganic carbon is necessary due to the inherent ability of carbonates to buffer acid inputs. If carbonates exist, they will be determined by manometric detection of evolved CO_2 after extraction with a strong acid, or by an automated CHN analyzer. Carbonates are not expected because the soils being sampled are generally thought to be acid sensitive. Inorganic carbon analyses will be performed only on soil samples reacting positively to a test for effervescence upon the addition of drops of 4 N HCl.

6.2.4 Extractable Sulfate - The amount of extractable sulfate will indicate the sulfate saturation of the anion exchange sites. Extractable sulfate is determined in two different extracts (DI water and 500 mg/L P). Extractable sulfate is then determined by ion chromatography. These analyses will be performed on all samples.

6.2.5 Sulfate Adsorption Isotherms - The ability of soil to adsorb sulfate is perhaps the most important parameter in determining if a soil unit will show direct or delayed response to added sulfate deposition. Isotherms will be developed by placing soil samples in six separate sulfate solutions for 1 hour and determining the amount adsorbed by analysis of the solution for sulfate after contact with the solution. These isotherms will represent the maximum sulfate adsorption capacity of the soil at the given conditions. Sulfate adsorption isotherms will not be required for organic horizons, but will be performed on all mineral horizons.

6.2.6 Total Sulfur - Total sulfur is measured because of its close relationship with extractable sulfate, and to inventory existing sulfur levels to monitor future inputs of anthropogenic sulfur. An automated method involving sample combustion followed by titration of evolved sulfur

6.2.7 Cation Exchange Capacity - Cation Exchange Capacity (CEC) is a standard soil characterization parameter and indicates the ability of the soil to adsorb exchangeable bases. Therefore, it is well correlated with soil buffering capacity. Ammonium chloride (NH_4Cl, pH 7.0), and ammonium acetate (NH_4OAc, pH 7.0), and 0.002 M calcium chloride ($CaCl_2$) will be used as the replacement solutions. The extractable bases (Na^+, K^+, Ca^{2+}, Mg^{2+}) will then be determined on the extracts by flame atomic absorption spectroscopy (AA) or inductively-coupled plasma atomic emission spectroscopy (ICP). These analyses will be performed on all samples.

6.2.8 Exchangeable Acidity - Exchangeable acidity is a measure of the remaining exchangeable soil cations that are not part of the base saturation. Two methods are specified. One employs a $BaCl_2$--triethanolamine extraction and the other employs a KCl extraction. The former extraction quantifies total exchangeable acidity and the latter quantifies effective exchangeable acidity. Aluminum acidity is also determined in the KCl extract by analyzing the extract for Al by AA or ICP. These analyses will be performed on all samples.

6.2.9 Extractable Iron and Aluminum - Iron oxides and aluminum oxides are highly correlated to sulfate adsorption and are important in standard soil characterization. Extractable Fe and Al are determined by AA or ICP in three different extracts. Each extract yields an estimate of a specific Al or Fe fraction. The three extracts (and fractions) are sodium pyrophosphate (organic Fe and Al), acid-oxalate (organic plus sesquioxides), and citrate-dithionite (nonsilicate Fe and Al). These analyses will be performed on all samples.

6.2.10 Lime and Aluminum Potential - Lime potential is used as an input for certain models instead of base saturation, it is defined as pH-1/2 pCa. Another characteristic shown to be important to watershed models is the relationship of pH to solution Al^{3+} levels, defined as the aluminum potential (K_A), which is 3pH-pAl. The method involves extracting the soil with 0.002 m $CaCl_2$ and determining pH, Ca, and Al in the extract. The remaining base cations, Na^+, K^+, and Mg^{2+}, as well as exchangeable Fe, will also be determined on this extract because of expediency and comparability to other extracts. These analyses will be performed on all samples.

7.0 BULK-DENSITY DETERMINATION

7.1 Scope

Bulk density is defined as the weight per unit volume of a soil. Bulk density generally ranges between 1.0 and 2.0 g/cm^3. For organic soils, bulk density commonly ranges from 0.050 to 0.355 g/cm^3. Bulk density will be determined by the field collection and Saran^{TM} coating of clods from each horizon, followed by weighing the clods by the preparation laboratories.

This method was chosen because of routine use in the field, relative ease of performance, and elimination of compaction problems inherent in core methods. It will be impossible to collect clods from certain horizons. Relationships between the particle-size distribution and surface area data and the pre-existing data may be used to derive values for missing data. The laboratory method was provided by Soil Morphology Laboratory, University of Massachusetts, Amherst, Massachusetts.

7.2 Apparatus and Materials

7.2.1 Dow Saran^{TM} S310 Resin - The Saran^{TM} resin dissolves readily in acetone or methylethyl ketone. Acetone is preferred and will be used because it is more readily available and less toxic.

7.2.2 Coating Solution - The coating solution will be prepared by the preparation laboratories and will be supplied to the field crews. To prepare the solution, calculate the amount of acetone required to make a 1:4 solution of resin to acetone. If a 1:7 solution is desired, the stock solution can be diluted with a precalculated volume of acetone. The resin is not readily soluble in acetone and will require mixing. Because the solvent is flammable, care should be taken during mixing. The solution should be made in an exhaust hood. A nonsparking electric stirrer should be used. If a high-speed stirrer is used, the resin dissolves in about 1 hour. If the solution must be made in the field, mix well and often with a wooden stick. Metal paint cans will be supplied as mixing containers, although other containers may be used as well. Some plastic containers are unsuitable because the acetone dissolves the plastic. Containers that can be tightly closed are most desirable because the solution is highly volatile and rapid evaporation will result in excesses of acetone being used. If the solution becomes thick, add more acetone until the desired consistency is reached.

7.3 Procedure

Collect natural clods (three per horizon) of about 100 cm^3 to 200 cm^3 in volume (approximately fist-size). Remove a piece of soil larger than the clod from the face of a sampling pit with a spade. From this piece, prepare a clod by gently cutting or breaking off protruding peaks and material sheared by the spade. If roots are present, they can be cut conveniently with scissors or side cutters. In some soils, clods can be removed directly from the face of the pit with a knife or spatula. No procedure for taking samples will fit all soils; the procedure must be adjusted to meet the conditions in the field at the time of sampling.

The clods are tied with fine copper wire or placed in hairnets and suspended from a rope or string, then hung out like a clothesline. The clods themselves should be labeled with some type of tag that can be attached to the hairnet or string. The label should record the sample code, horizon, and replicate number. Moisten clods with a fine mist spray. The suspended clods are dipped by raising a container of the dipping mixture upward to submerse each clod momentarily. The number of times a clod is dipped should be recorded on the label. The saran-coated clods should be allowed to dry for 30 minutes or longer.

7.3.1 Transport of Clods - Clods should be sealed in the presupplied 6" X 8" plastic bags, then placed in the compartmentalized clod boxes. The top (inner face) of the clod box should be labeled with the same information on the clod tag (i.e., sample code, horizon, replicate number, and how many times the clod was dipped in the Saran^{TM}). Great care must be taken to ensure that the clods are not broken or damaged during handling and shipping. Space not occupied by the clods in each compartment should be filled with packing material, for example, leaves, newspaper, or extra plastic bags. Clod boxes may be reused by removing the old labels.

7.3.2 Preparation Laboratory Handling of Clods - Upon receipt of clods, labels should be removed and placed in the Bulk Density Preparation Laboratory Notebook. However, the clods must be relabeled with the appropriate sample number to retain identity. Notes should be made in the notebook regarding the condition of the clod upon arrival, how many times the clod was dipped in Saran^{TM} in the field, label clarity, and the time of receipt. At the end of the project, this notebook should be submitted to Lockheed-EMSCO (EPA EMSL-LV) Data Audit Supervisor

7.3.3 Bulk-density Procedure

7.3.3.1 Weigh the clod and record this weight in the laboratory notebook as m_1.

7.3.3.2 Dip the clod briefly in a saran:acetone (1:6 w/w) solution and allow the coating to dry.

7.3.3.3 Reweigh the clod and record this weight as m_2.

7.3.3.4 Repeat steps 7.3.3.2 and 7.3.3.3 as needed to obtain an impervious coating. Record weights after each coating as m_3, m_4, etc.

7.3.3.5 Place a 1-L beaker that contains 600 to 700 mL of deaired and distilled water of known temperature (recorded as T) on balance pan and record the tare weight as M_A.

7.3.3.6 Suspend the clod over the beaker, lower it gently into the water until totally submerged, then record the weight displayed on the balance as M_B.

7.3.3.7 Suspend the clod in a convection oven at 105°C for 48 hours.

7.3.3.8 Remove the clod from the oven, weigh it, and record this weight as M_{OD}.

7.3.3.9 Place the clod in an appropriate container and put the container into an electric muffle furnace for 2 hours at 400°C.

7.3.3.10 After the sample has cooled, weigh the contents of the container and record this as m_t.

7.3.3.11 Pass the sample through a 2-mm sieve and obtain the weights of coarse fragments and the fine-earth fraction. Record these as M_{CF} and m_{fc}, respectively.

7.3.3.12 Calculations

$$BD_{FM} = \frac{M_{OD} - [M_{CF} + M_{TS}\,(0.85)]}{\frac{M_V}{r\,H_2O_T} - \frac{M_{CF}}{2.65} + \frac{M_{TS}}{1.30}}$$

where BD_{FM} is the field moist bulk density

M_{OD} is the oven-dry weight of the clod (Step 7.3.3.8)

M_{CF} is the weight of the coarse fragments in the clod (Step 7.3.3.11)

M_{TS} is the weight of the air-dry Saran^{TM} coating which may be estimated as follows:

$$M_{TS} = \frac{X\,(m_a - m_1)}{a-1}$$

where X is the total number of coatings (field + lab)

a is the number of laboratory coatings

m_a is the clod weight after the final coating

m_1 is the initial clod weight after unpacking

M_V is equal to $M_B - M_A$ (from steps 7.3.3.5 and 7.3.3.6)

$r\,H_2O_T$ is the density of water obtained from Table 7.1 for the temperatures measured in Step 7.3.3.5.

The final value to be reported on Form 101 is the coarse-fragment, and Saran^{TM}-weight corrected value.

7.3.3.13 Assumptions

Four assumptions are made concerning the bulk-density procedure:

- The weight of the individual, field-applied Saran^{TM} coatings is equivalent to that applied in the laboratory, and the Saran^{TM} has not infiltrated the clod.
- The specific gravity of the coarse fragments is 2.65.
- The specific gravity of air-dried Saran^{TM} is 1.30.
- The Saran^{TM} loses 15 percent of its weight upon oven drying at 105°C for 48 hours.

TABLE 7.1. SPECIFIC GRAVITY* OF WATER

°C	0	1	2	3	4	5	6	7	8	9
0	0.9999	0.9999	1.0000	1.0000	1.0000	1.0000	1.0000	0.9999	0.9999	0.9999
10	0.9997	0.9996	0.9995	0.9994	0.9993	0.9991	0.9990	0.9988	0.9986	0.9984
20	0.9982	0.9980	0.9978	0.9976	0.99973	0.9971	0.9968	0.9965	0.9963	0.9960
30	0.9957	0.9954	0.9951	0.9947	0.9944	0.9941	0.9937	0.9934	0.9930	0.9926
40	0.9922	0.9919	0.9915	0.9911	0.9907	0.9902	0.9898	0.9894	0.9890	0.9885
50	0.9881	0.9876	0.9872	0.9867	0.9862	0.9857	0.9852	0.9848	0.9842	0.9838
60	0.9832	0.9827	0.9822	0.9817	0.9811	0.9806	0.9800	0.9795	0.9789	0.9784
70	0.9778	0.9772	0.9767	0.9761	0.9755	0.9749	0.9743	0.9737	0.9731	0.9724
80	0.9718	0.9712	0.9706	0.9699	0.9693	0.9686	0.9680	0.9673	0.9667	0.9660
90	0.9653	0.9647	0.9640	0.9633	0.9626	0.9619	0.9612	0.9605	0.9598	0.9591

* Also the density or unit weight of water in grams per milliliter.

8.0 CREWS, SUPPLIES, AND EQUIPMENT

8.1 Scope

Field crews will consist of four SCS employees. The lead soil scientist in each crew will supervise all field operations. This person will be responsible for selecting each sampling site in the field and for documentating of all field data. The following is a list of supplies needed for each field crew.

- 35-mm camera (macro lens or wide-angle lens)
- ASA-400 film and Kodak premailer envelopes
- 2 clinometers
- Munsell color charts
- Magnetic compass
- Hand lens
- 2 brass sieves (3/4", 10 mesh, 19 mm)*

- 2 thermometers* (centigrade)
- 5 coolers*
- 40 Blue Ice™ gel packs*
- Stereoscope
- 0.1 N HCL or 10% 4 N HCL and drop bottle
- Visqueen™ 6-mil sheets, (4'x4')*
- Spring scale (optional; use an exterior canvas bag for weighing)
- Plastic inner sample bags (20/day)*
- Canvas exterior sample bags (20/day)*
- NADSS Label A (30/day)*
- Orange flagging (1 roll/day)*
- Yellow marker flags (20/day)*

- 5 indelible-ink markers*

- SCS Form SOI 232 and clipboard

- Field logbook*

- 1-gallon paint can with lid*

- Saran^{TM}* and acetone (Note: acetone must be purchased locally)

- Hair nets (1 per clod)*

- 6"x8" plastic bags, 1 mil (enough for one per clod)*

- 24-cell, 17.50"x11.94"x3.75" boxes (1 box per day - reusable)*

- 2"x2" blank vinyl labels (attach to box for individualized clod compartments)*

- Hand auger (for sampling Histosols; optional, may use spades)

- Staplers*

- Saran^{TM} Dow-310 resin*

An asterisk indicates that the item will be shipped by EPA EMSL-LV. The amount of equipment sent to each preparation laboratory is based on the number of crews assigned to that laboratory.

The crews from New York and Pennsylvania (4) will receive supplies from the Cornell University soil preparation laboratory. Maine crews (2) will receive supplies from the University of Maine at Orono soil preparation laboratory. Rhode Island-Connecticut (1), New Hampshire (1), and Massachusetts crews will receive supplies from the University of Massachusetts at Amherst, Massachusetts.

8.2 Equipment Notes

8.2.1 Coolers and Gel Packs - For each day of sampling, five coolers and eight gel packs per cooler should be stored in the field sampling vehicle. The gel packs should be frozen in advance. Enough frozen gel packs should be stored in a storage cooler to replace softened gel packs if ambient temperature in the cooler falls below 4°C. Coolers containing gel packs and soil samples should be taped shut before transit. Two thermometers per crew will be provided for routine temperature checks on coolers containing gel packs and soil samples. Temperature readings to the nearest tenth of a degree should be recorded in the field notebook. Time and date should also be recorded in the notebook.

8.2.2 Marker Flags and Flagging - Upon arrival at the sample site, orange flagging should be tied to surrounding shrubbery at eye level. This flagging is necessary in case of return visit to the pedon. The 21 inch stake yellow flags should be placed at least 6 inches into the ground at the four corners of the pedon before leaving the sample site.

8.2.3 Visqueen^{TM} Plastic Sheets - Visqueen^{TM} plastic sheets (4'x4', 6 mil) will be provided for each crew. All soil materials less than 20 mm should be sieved onto these sheets. The sample is then poured into the inner plastic prelabeled sample bag. If by visual estimate the 2 to 20 mm particle-size class exceeds 50 percent by volume, two 5.5-kg samples should be bagged and sieved for that sample. A canvas sheet may be substituted for the 4'x4' plastic sheet, but the use of this should be noted in the field notebook and should be immediately reported to the EPA EMSL-LV QA officer

8.2.4 Field Notebook - Daily activities of the field crew should be logged in a field notebook. Each day's activities should be recorded, specific problems, solutions, and other miscellaneous notes should be recorded, along with location and identification of each sample pedon. These field notebooks will be submitted to Lockheed-EMSCO (EPA-EMSL-LV) in care of:

Lockheed Engineering and Management
Services Company, Inc.
1050 E. Flamingo, Suite 120
Las Vegas, Nevada 89109

9.0 REFERENCES

1. USDA/SCS. 1983. National Soils Handbook. Part 600-606. U.S. Government Printing Office, Washington D.C.

2. USDA/SCS. 1984. SCS National Soil Survey Manual. U.S. Government Printing Office, Washington D.C.

3. Mausbach, M., R. Yeck, D. Nettleton, and W. Lynn. 1983. Principles and Procedures for Using Soil Survey Laboratory Data. National Soil Survey Laboratory. Lincoln, Nebraska.

4. USDA/SCS. 1984. Soil Survey Laboratory Methods and Procedures for Collecting Soil Samples. Soil Survey Investigations Report No. 1. U.S. Government Printing Office, Washington D.C.

APPENDIX A

FIELD DATA FORMS AND LEGENDS

1.0 FORM SCS-SOI-232

SOIL DESCRIPTION

10-84

(SEE BACK PAGE FOR SITE DESCRIPTION CODES)

SERIES NAME	SAMPLE NUMBER ST COUNTY UNIT SUB	MLRA SUB	LATITUDE DEG MIN SEC DIR	LONGITUDE DEG MIN SEC DIR	DATE MO YEAR

SLOPE % SHP GAI ASP	MICRO E A F POS	PHYS	PEDON CLASSIFICATION (See Page 5 of Instructions for codes) O SO GG SG PSC MIN RE TMP OTH	PRECIP CM	WATER TABLE DEPTH MONTH CM SD	LU	ST	PM

ELEVATION METERS	PARENT MATERIAL (See Page 8 of Instructions for codes) 1 W M ORG | 2 W M ORG | 3 W M ORG | 4 W M ORG	BORR	TEMPERATURES °C AVERAGE AIR: ANN SUM WINTER AVERAGE SOIL: ANN SUM WINTER	MST RGE	WEATHER STATION NUMBER

CONTROL SECTION CM	ER WA	AN OT	DIAGNOSTIC FEATURES DEPTH CM END | DEPTH CM END | DEPTH CM END | DEPTH CM END | DEPTH CM END	FLOODING FRQ DUR

VEGETATION SPECIES MAJOR 2ND 3RD	DESCRIBERS NAMES

LOCATION DESCRIPTION & FREE FORM SITE NOTES

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FORM SCS-SOI-232 (Continued)

	DEPTH		HORIZON DESIGNATION			VOL	THICKNESS			DRY COLOR					MOIST COLOR					TEXTURE	
	UPPER	LOWER	DISC	MASTER LETTER	SUFFIX	EAT / TOT	AVE	MAX	MIN	LOC	%	HUE	VAL	CHR	LOC	%	HUE	VAL	CHR	CLASS	MOD
1																					
2																					
3																					
4																					
5																					
6																					
7																					
8																					
9																					
10																					

page 2 of 4

FORM SCS-SOI-232 (Continued)

	STRUCTURE			CONSISTENCE			MOTTLES						SURFACE FEATURES									EFFERVESCENCE			FIELD MEASURED PROPERTIES		
	GRD	SZ	SHP	DRY MOIST OTHER	ST/PL	CEM	AB	SZ	CON	HUE	VAL	CHR	KND	AMT	CN	DST	LOC	HUE	VAL	CHR	BOUNDARY	CL	AG	EX	KND	AMOUNT	°.
1																											
2																											
3																											
4																											
5																											
6																											
7																											
8																											
9																											
10																											

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FORM SCS-SOI-232 (Continued)

	ROOTS			PORES			CONCENTRATIONS				ROCK FRAGMENTS			FREE FORM HORIZON NOTES
	QT	SZ	LOC	SHP	QT	SZ	KND	QT	SHP	SZ	KND	%	SZ	
1														
2														
3														
4														
5														
6														
7														
8														
9														
10														

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2.0 SOIL DESCRIPTION CODES FOR FORM SCS-SOI-232

2.1 Great Group Codes

ALFISOLS

AAQAL	Albaqualf
AAQNA	Natraqualf
AAQPN	Plinthaqualf
ABOCR	Cryoboralf
ABOGL	Glossoboralf
AUDAG	Agrudalf
AUDFS	Fraglossudalf
AUDNA	Natrudalf
AUSDU	Durustalf
ASUPA	Paleustalf
AXEDU	Durixeralf
AXENA	Natrixeralf
AXERH	Rhodoxeralf
AAQDU	Duraqualf
AAQGL	Glossaqualf
AAQTR	Tropaqualf
ABOEU	Eutroboralf
ABONA	Natriboralf
AUDFE	Ferrudalf
AUDGL	Glossudalf
AUDPA	Paleudalf
AUSHA	Haplustalf
AUSPN	Plinthustalf
AXEFR	Fragixeral
AXEPA	Palexeralf
AAQFR	Fragiaqualf
AAQOC	Ochraqualf
AAQUM	Umbraqualf
ABOFR	Fragiboralf
ABOPA	Paleboralf
AUDFR	Fragiudalf
AUDHA	Hapludalf
AUDTR	Tropudalf
AUSNA	Natrustalf
AUSRH	Rhodustalf
AXEHA	Haploxeralf
AXEPN	Plinthoxeralf

ARIDISOLS

DARDU	Durargid
DARNT	Natrargid
DORCM	Camborthid
DORPA	Paleorthid
DARHA	Haplargid
DARPA	Paleargid
DORDU	Durorthid
DORSA	Salorthid
DARND	Nadurargid
DORCL	Calciorthid
DORGY	Gypsiorthid

ENTISOLS

EAQCR	Cryaquent
EAQHY	Hydraquent
EAQTR	Tropaquent
EFLTO	Torrifluvent
EFLUS	Ustifluent
EORTO	Torriorthent
EORUS	Ustorthent
EPSQU	Quartzipsamment
EPSUD	Udipsamment
EAQFL	Fluvaquent
EAQPS	Psammaquent
EARAR	Arent
EFLTR	Tropofluvent
EFLXE	Xerofluvent
EORTR	Troporthent
EORXE	Xerorthent
EPSTO	Torripsamment
EPSUS	Ustipsamment
EAQHA	Haplaquent
EAQSU	Sulfaquent

EFLCR Cryofluvent
EFLUD Udifluvent
EORCR Cryorthent
EORUD Udorthent
EPSCR Cryopsamment
EPSTR Tropopsamment
EPSXE Xeropsamment

HISTOSOLS

HFIBO Borofibrist
HFIME Medifibrist
HFOBO Borofolist
HHEBO Borohemist
HHEME Medihemist
HHETR Tropohemist
HSAME Medisaprist
HFICR Cryofibrist
HFISP Sphagnofibrist
HFOCR Cryofolist
HHECR Cryohemist
HHESI Sulfihemist
HSABO Borosaprist
HSATR Troposaprist
HFILU Luvifibrist
HFITR Tropofibrist
HFOTR Tropofolist
HHELU Luvihemist
HHESO Sulfohemist
HSACR Cryosaprist

INCEPTISOLS

IANCR Cryandept
IANEU Eutrandept
IANVI Vitrandepth
IAQFR Fragiaquept
IAQHU Humaquept
IAQSU Sulfaquept
IOCDU Durochrept
IOCFR Fragiochrept
IPLPL Plaggept
ITRHU Humitropept
IUMCR Cryumbrept
IUMXE Xerumbrept
IANDU Durandept
IANHY Hydrandept
IAQAN Andaquept
IAQHL Halaquept
IAQPK Palacaquept
IAQTR Tropaquept
IOCDY Dystrochrept
IOCUS Ustochrept
ITRDY Dystropept
ITRSO Sombritropept
IUMFR Fragiumbrept
IANDY Dystrandept
IANPK Placandept
IAQCR Cryaquept
IAQHP Haplaquept
IAQPN Plinthaquept
IOCCR Cryochrept
IOCEU Eutrochrept
IOCXE Xerochrept
ITREU Eutropept
ITRUS Ustropept
IUMHA Haplumbrept

MOLLISOLS

MALAR Argialboll
MAQCA Calciaquoll
MAQHA Haplaquoll
MBOCA Calciboroll
MBONA Natriboroll
MRERE Rendoll
MUDPA Paleudoll
MUSCA Calciustoll
MUSNA Natrustoll
MXEAR Argixeroll
MXEHA Haploxeroll
MALNA Natralboll
MAQCR Cryaquoll
MAQNA Natraquoll

MBOCR	Cryoboroll
MBOPA	Paleboroll
MUDAR	Argiudoll
MUDVE	Vermudoll
MUSDU	Durustoll
MUSPA	Paleustoll
MXECA	Calcixeroll
MXENA	Natrixeroll
MAQAR	Argiaquoll
MAQDU	Duraquoll
MBOAR	Argiboroll
MBOHA	Haploboroll
MBOVE	Vermiboroll
MUDHA	Hapludoll
MUSAR	Argiustoll
MUSHA	Haplustoll
MUSVE	Vermustoll
MXEDU	Durixeroll
MXEPA	Palexeroll

OXISOLS

OAQGI	Givvsiaquox
OAQUM	Umbraquox
OHUHA	Haplohumox
OOREU	Eutrorthox
OORSO	Sombriorthox
OUSAC	Acrustox
OUSHA	Haplustox
OAQOC	Ochraquox
OHUAC	Acrohumox
OHUSO	Sombrihumox
OORGI	Gibbsiorthox
OORUM	Umbriorthox
OUSEU	Eutrustox
OUSSO	Sombriustox
OAQPN	Plinthaquox
OHUGI	Gibbsihumox
OORAC	Acrorthox
OORHA	Haplorthox
OTOTO	Torrox

SPODOSOLS

SAQCR	Cryaquod
SAQHA	Haplaquod
SAQTR	Tropaquod
SHUFR	Fragihumod
SHUTR	Tropohumod
SORHA	Haplorthod
SAQDU	Duraquod
SAQPK	Placaquod
SFEFE	Ferrod
SHUHA	Haplohumod
SORCR	Cryorthod
SORPK	Placorthod
SAQFR	Fragiaquod
SAQSI	Sideraquod
SHUCR	Cryohumod
SHUPK	Placohumod
SORFR	Fragiorthod
SORTR	Troporthod

ULTISOLS

UAQAL	Albaquult
UAQPA	Paleaquult
UAQUM	Umbraquult
UHUPN	Plinthohumult
UUDFR	Fragiudult
UUDPN	Plinthudult
UUSHA	Haplustult
UUSRH	Rhodustult
UAQFR	Fragiaquult
UAQPN	Plinthaquult
UHUHA	Haplohumult
UHUSO	Sombrihumult
UUDHA	Hapludult
UUDRH	Rhodudult
UUSPA	Paleustult
UXEHA	Haploxerult
UAQOC	Ochraquult
UAQTR	Tropaquult

UHUPA	Palehumult
UUDTR	Tropudult
UHUTR	Tropohumult
UUSPN	Plinthustult
UUDPA	Paleudult
UXEPA	Palexerult

VERTISOLS

VTOTO	Torrert
VUSPE	Pellustert
VUSCH	Chromustert
VUDPE	Pelludert
VXEPE	Pelloxerert
UXECH	Chromxerert
VUDCH	Chromudert

2.2 Subgroup Codes

AA	Typic
AB	Abruptic
AB04	Abruptic aridic
AB08	Abruptic cryic
AB10	Abruptic haplic
AB14	Abruptic ultic
AB16	Abruptic xerollic
AE	Aeric
AE03	Aeric arenic
AE05	Aeric grossarenic
AE06	Aeric humic
AE08	Aeric mollic
AE09	Aeric tropic
AE10	Aeric umbric
AE12	Aeric xeric
AL	albaquic
AL02	Albaquultic
AL04	Albic
AL08	Albic glossic
AL10	Alfic
AL13	Alfic andeptic
AL12	Alfic arenic
AL16	Alfic lithic
AN24	Andaqueptic
AN01	Andeptic
AN11	Andeptic glossoboric
AN	Andic
AN06	Andic Dystric
AN22	Andic ustic
AN30	Anthropic
AQ	Aqualfic
AQ02	Aquentic
AQ04	Aqueptic
AQ06	Aquic
AQ08	Aquic arenic
AQ14	Aquic duric
AQ16	Aquic duriorthidic
AQ18	Aquic dystric
AQ24	Aquic haplic
AQ26	Aquic lithic
AQ31	Aquic psammentic
AQ34	Aquollic
AQ36	Aquultic
AR	Arenic
AR02	Arenic aridic
AR03	Arenic orthoxic
AR04	Arenic plinthaquic
AR06	Arenic plinthic
AR08	Arenic rhodic
AR10	Arenic ultic
AR14	Arenic umbric
AR16	Arenic ustalfic
AR18	Arenic ustollic
AR22	Argiaquic
AR24	Argiaquic xeric
AR26	Argic
AR28	Argic lithic
AR30	Argic pachic
AR32	Argic vertic
AR34	Aridic
AR36	Aridic calcic
AR42	Aridic duric
AR50	Aridic pachic
AR52	Aridic petrocalcic
AN03	Andaquic
BO	Boralfic

BO02	Borolfic lithic	BO04	Boroalfic udic
BO06	Borollic	BO08	Borollic glossic
BO10	Borollic lithic	BO12	Borollic vertic
CA	Calcic	CA04	Calcic pachic
CA06	Calciorthidic	CA10	Calcixerollic
CA20	Cambic	CH	Chromic
CH06	Chromudic	CR	Cryic
CR10	Cryic lithic	CR14	Cyric pachic
CU	Cumulic	CU02	Cumulic udic
CU04	Cumulic ultic		
DU	Durargidic	DU02	Duric
DU08	Durixerollic	DU10	Durixerollic lithic
DU11	Durochreptic	DU12	Durorthidic
DU14	Durorthidic xeric	DY02	Dystric
DY03	Dystric entic	DY04	Dystric Fluventic
DY06	Dystric lithic	DY08	Dystropeptic
EN	Entic	EN02	Entic lithic
EN06	Entic ultic	EP	Epiaquic
EP10	Epiaquic orthoxic	EU	Eutric
EU02	Eutrochreptic	EU04	Eutropeptic
FE	Ferrudalfic	FI	Fibric
FI02	Fibric terric	FL02	Fluvaquentic
FL06	Fluventic	FL12	Fluventic umbric
FR10	Fragiaquic	FR18	Fragic
GL02	Glossaquic	GL04	Glossic
GL10	Glossic udic	GL12	Glossic ustollic
GL14	Glossoboralfic	GL16	Glossoboric
GR	Grossarenic	GR01	Grossarenic entic
GR04	Grossarenic plinthic		
HA01	Haplaquic	HA	Haplaquodic
HA02	Haplic	HA05	Haplohumic
HA07	Haploxerollic	HA09	Hapludic
HA12	Hapludollic	HA16	Haplustollic
HE	Hemic	HE02	Hemic terric
HI	Histic	HI02	Histic lithic
HI06	Histic pergelic	HU10	Humaqueptic
HU	Humic	HU02	Humic lithic
HU05	Humic pergelic	HU06	Humoxic
HY	Hydric	HY02	Hydric lithic
LE	Leptic	LI	Limnic

LI01	Lithic	LI04	Lithic mollic
LI06	Lithic ruptic-alfic	LI07	Lithic ruptic-argic
LI09	Lithic ruptic-entic	LI08	Lithic ruptic-entic-xerollic
LI13	Lithic ruptic-ultic	LI15	Lithic ruptic-xerochreptic
LI11	Lithic ruptic-xerorthentic	LI10	Lithic udic
LI12	Lithic ultic	LI14	Lithic umbric
LI16	Lithic ustic	LI18	Lithic ustollic
LI20	Lithic vertic	LI22	Lithic xeric
LI24	Lithic xerollic		
MO	Mollic	NA06	Natric
OC	Ochreptic	OR01	Orthic
OR	Orthidic	OR02	Orthoxic
OX	Oxic		
PA	Pachic	PA02	Pachic udic
PA04	Pachic ultic	PA06	Paleorthidic
PA08	Paleustollic	PA10	Palexerollic
PA20	Paralithic vertic	PE	Pergelic
PE01	Pergelic ruptic-histic	PE02	Pergelic sideric
PE04	Petrocalcic	PE06	Petrocalcic ustalfic
PE08	Petrocalcic ustollic	PE14	Petrocalcic xerollic
PE16	Petroferric	PE20	Petrogypsic
PK	Placic	PK10	Plaggeptic
PK12	Plaggic	PL	Plinthaquic
PL04	Plinthic	PL06	Plinthudic
PS	Psammaquentic	PS02	Psammentic
QU	Quartzipsammentic		
RE	Rendollic	RH	Rhodic
RU02	Ruptic-alfic	RU09	Ruptic-lithic
RU11	Ruptic-lithic-entic	RU15	Ruptic-lithic-xerochreptic
RU17	Ruptic-ultic	RU19	Ruptic-vertic
SA	Salorthidic	SA02	Sapric
SA04	Sapric terric	SI	Sideric
SO04	Sombrihumic	SP	Sphagnic
SP02	Sphagnic terric	SP04	Spodic
SU	Suflic		
AA	Typic	TE	Terric
TH04	Thapto-histic	TH06	Thapto-histic tropic
TO	Torrertic	TO02	Torrifluventic
TO04	Torriorthentic	TO06	Torripsammentic
TO10	Torroxic	TR	Tropaquodic

TR02	Tropeptic	TR04	Tropic
UD01	Udalfic	UD	Udertic
UD02	Udic	UD03	Udollic
UD05	Udorthentic	UD10	Udoxic
UL	Ultic	UM	Umbreptic
UM02	Umbric	US	Ustalfic
US02	Ustertic	US04	Ustic
US06	Ustochreptic	US08	Ustollic
US12	Ustoxic		
VE	Vermic	VE02	Vertic
XE	Xeralfic	XE02	Xerertic
XE04	Xeric	XE08	Xerollic

2.3 Slope Shape Codes

1 convex	2 plane	3 concave	4 undulating	5 complex

2.4 Geomorphic Position Codes

01 summit crested hills	11 summit interfluve
02 shoulder crested hills	12 shoulder interfluve
22 shoulder headslope	42 shoulder noseslope
03 backslope crested hills	23 backslope headslope
33 backslope sideslope	43 backslope noseslope
24 footslope headslope	34 footslope sideslope
44 footslope noseslope	05 toeslope crested hills
25 toeslope headslope	35 toeslope sideslope

2.5 Slope Aspect Codes

1 northeast	2 east	3 southeast	4 south
5 southwest	6 west	7 northwest	8 north

2.6 Pedon Position Codes

1 on the crest	3 on upper third	2 on slope and crest
4 on middle third	5 on lower third	6 on a slope
7 on a slope and depression	9 in a drainageway	
8 in a depression		

2.7 Regional Landform Codes

A coastal plains	B intermountain basin
E lake plains	F river valley

G glaciated uplands
I bolson
M mountains or deeply disected plateaus
N high hills
R hills
V mountain valleys or canyons
H glaciofluvial landform
L level or undulating uplands
P piedmonts
U plateaus or tablelands

2.8 Local Landform Codes

AA depression
B bog
D dome or volcanic cone
F broad plain
H abandoned channel
J moraine
L drumlin
N low sand ridge--nondunal
Q playa or alluvial flat
S sand dune or hill
U terrace--outwash or marine
W swamp or marsh
Y barrier bar
A fan
C cuesta or hogback
E escarpment
G crater
I hillside or mountainside
K kamefield
M mesa or butte
P flood plain
R upland slope
T terrace--stream or lake
V pediment
X salt marsh
Z back barrier flat

2.9 Particle Size Codes

002 not used

005 ashy
008 ashy over loamy
019 ashy over medial
007 ashy over cindery
013 ashy over loamy-skeletal
009 ashy-skeletal

003 cindery
015 cindery over medial-skeletal
004 cindery over sandy or sandy-skeletal
006 cindery over loamy

114 clayey
116 clayey over fragmental
120 clayey over loamy-skeletal
118 clayey over sandy or sandy-skeletal
056 clayey-skeletal
122 clayey over fine-silty
124 clayey over loamy
058 clayey-skeletal over sandy

080 coarse-loamy
082 coarse-loamy over fragmental
084 coarse-loamy over sandy or sandy-skeletal
088 coarse-silty
090 coarse-silty over fragmental
092 coarse-silty over sandy or sandy-skeletal
086 coarse-loamy overy clayey
094 coarse-silty over clayey

126 fine
096 fine-loamy
102 fine-loamy over clayey
098 fine-loamy over fragmental
100 fine-loamy over sandy or sandy-skeletal
106 fine-silty
112 fine-silty over clayey
108 fine-silty over fragmental
110 fine-silty over sandy or sandy-skeletal

036 fragmental
068 loamy
072 loamy over sandy or sandy-skeletal
050 loamy-skeletal
054 loamy-skeletal over clayey
051 loamy-skeletal over fragmental
052 loamy-skeletal over sand
010 medial
012 medial over cindery
014 medial over clayey
016 medial over fragmental
018 medial over loamy
020 medial over loamy-skeletal
022 medial over sandy or sandy-skeletal
024 medial over thixotropic
011 medial-skeletal

062 sandy
063 sandy or sandy-skeletal
066 sandy over clayey
064 sandy over loamy
044 sandy-skeletal
046 sandy-skeletal over loamy
047 sandy-skeletal over clayey

026 thixotropic
028 thixotropic over fragmental
034 thixotropic over loamy
032 thixotropic over loamy-skeletal
030 thixotropic over sandy or sandy-skeletal
027 thixotropic-skeletal
134 very fine

2.10 Mineralogy Codes

02 not used
04 calcareous
05 carbonatic
09 chloritic
07 clastic
08 coprogenous
10 diatomaceous
12 ferrihumic
14 ferritic
18 gibbsitic
20 glauconitic
22 gypsic
24 halloysitic
26 illitic
27 illitic (calcareous)
28 kaolinitic
30 marly
32 micaceous
34 mixed
35 mixed (calcareous)
37 montmorillonitic
38 montmorillonitic (calcareous)
40 oxidic
42 sepiolitic
44 serpentinitic
46 siliceous
50 vermiculitic

2.11 Reaction Codes

02 not used
04 acid
08 dysic
10 euic
12 nonacid
14 noncalcareous

2.12 Temperature Regime Codes

02 not used   04 frigid   06 hyperthermic   08 isofrigid
10 isohyperthermic   12 isomesic   14 isothermic   16 mesic
18 thermic

2.13 Other Family Codes

02 not used   04 coated   05 cracked   06 level
08 micro   12 ortstein   14 shallow
15 shallow and coated   17 shallow and uncoated
16 sloping   20 uncoated

2.14 Kind of Water Table Codes

1 flooded   2 perched   3 apparent   4 ground

2.15 Landuse Codes

C cropland   I cropland irrigated
E forest land grazed   F forest land not grazed
G pasture land and native pasture   H horticultural land
L waste disposal land   N barren land
P rangeland grazed   S rangeland not grazed
R wetlands   Q wetlands drained
T tundra   U urban and built-up land

2.16 Permeability Codes

1 very slow   2 slow   3 moderately slow   4 moderate
5 moderately rapid   6 rapid   7 very rapid

2.17 Drainage Codes

1 very poorly drained   2 poorly drained
3 somewhat poorly drained   4 moderately well drained
5 well drained   6 somewhat excessively drained
7 excessively drained

2.18 Parent Material Weathering Codes

1 slight   2 moderate   3 high

2.19 Parent Material Mode of Deposition Codes

A alluvium   E eolian   H volcanic ash   W loess
S eolian-sand   D glacial drift   G glacial outwash   T glacial till

L lacustrine
V local colluvium
R solid rock
M marine
U unconsolidated sediments
O organic
X residuum
Y solifluctate

2.20 Parent Material Origin Codes

Mixed Lithology

Y0 mixed
Y2 mixed-calcareous
Y4 mixed-igneous-metamorphic and sedimentary
Y6 mixed-igneous and sedimentary
Y1 mixed-noncalcareous
Y3 mixed
Y5 mixed-igneous and metamorphic
Y7 mixed-metamorphic and sedimentary

Conglomerate

C0 conglomerate
C2 conglomerate-calcareous
C1 conglomerate-noncalcareous

Igneous

10 igneous
11 igneous-coarse
13 igneous-intermediate
15 igneous-fine
17 igneous-andesite
19 igneous-ultrabasic
12 igneous-basic
14 igneous-granite
16 igneous-basalt
18 igneous-acid

Metamorphic

M0 metamorphic
M1 gneiss
M3 metamorphic-basic
M5 schist and thyllite
M7 metamorphic-basic
M9 quartzite
M2 metamorphic-acidic
M4 serpentine
M6 metamorphic-acidic
M8 slate

Sedimentary

S0 sedimentary
S1 marl
S2 glauconite

Interbedded Sedimentary

B0 interbedded sedimentary
B2 limestone-sandstone
B4 limestone-siltstone
B1 limestone-sandstone-shale
B3 limestone-shale
B5 sandstone-shale

B6 sandstone-siltstone
B7 shale-siltstone

Sandstone

A0 sandstone
A2 arkosic-sandstone
A4 sandstone-calcareous
A1 sandstone-noncalcareous
A3 other sandstone

Shale

H0 shale
H1 shale-noncalcareous
H2 shale-calcareous

Siltstone

T0 siltstone
T2 siltstone-calcareous
T1 siltstone-noncalcareous

Limestone

L0 limestone
L1 chalk
L3 dolomite
L5 limestone-arenaceous
L7 limestone-cherty
L2 marble
L4 limestone-phosphatic
L6 limestone-argillaceous

Pyroclastic

P0 pyroclastic
P1 tuff
P3 tuff-basic
P5 breccia-acidic
P7 tuff-breccia
P9 pahoehoe
P2 tuff-acidic
P4 volcanic breccia
P6 breccia-basic
P8 aa

Ejecta Material

E0 ejecta-ash
E1 acidic-ash
E3 basaltic-ash
E5 cinders
E7 scoria
E2 basic-ash
E4 andesitic-ash
E6 pumice
E8 volcanic bombs

Organic Materials

K0 organic
K2 herbaceous material
K1 mossy material
K3 woody material

K4 wood fragments
K6 charcoal
K9 other organics
K5 logs and stumps
K7 coal

2.21 Moisture Regime Codes

AR aridic moisture regime
US ustic moisture regime
AQ aquic moisture regime
XE xeric moisture regime
UD udic moisture regime
TO torric moisture regime
PU perudic moisture regime

2.22 Erosion Codes

1 slight
0 none
2 moderate
3 severe

2.23 Runnoff Codes

0 none
5 rapid
1 ponded
4 moderate
2 very slow
6 very rapid
3 slow

2.24 Diagnostic Feature Codes

A anthropic
O ochric
D durinodes
W paralithic contact
T argillic
G gypsic
E petrocalcic
Y salic
V sulfuric
H histic
P plaggen
Z duripan
Q albic
C calcic
N natric
J petrogypsic
I sombric
F fragipan
M mollic
U umbric
L lithic contact
R argic
B cambic
X oxic
K placic
S spodic

2.25 Horizon Codes

Color Location Codes

0 unspecified
1 ped interior
2 ped exterior
3 rubbed or crushed

Texture Classes

C clay
CL clay loam
COSL coarse sandy loam
FM fragmental material
FSL fine sandy loam
L loam
LFS loamy fine sand
CIND cinders
COS coarse sand
CSCL coarse sandy clay loam
FS fine sand
G gravel
LCOS loamy coarse sand
LS loamy sand

LVFS	loamy very fine sand	S	sand
SC	sandy clay	SCL	sandy clay loam
SG	sand and gravel	SI	silt
SIC	silty clay	SICL	silty clay loam
SIL	silt loam	SL	sandy loam
VFS	very fine sand	VFSL	very fine sandy loam
ICE	ice or frozen soil	GYP	gypsiferous earth
VAR	variable	CE	coprogenous earth
DE	diatomaceous earth	FB	fibric material
MARL	marl	MPT	mucky peat
MUCK	muck	PDOM	partially decomposed organics
UDOM	undecomposed organics	PEAT	peat
SP	sapric material		
OPWD	oxide protected weathered bedrock		
U	unknown texture	UWB	unweathered bedrock
WB	weathered bedrock		

Texture Modifiers

BY	bouldery	BYV	very bouldery	BYX	extremely bouldery
ST	stony	STV	very stony	STX	extremely stony
CB	cobbly	CBA	angular cobbly	CBV	very cobbly
CBX	extremely cobbly	CN	channery	SNV	very channery
CNX	extremely channery	CR	cherty	CRC	coarse cherty
CRV	very cherty	CRX	extremely cherty	FL	flaggy
FLV	very flaggy	FLX	extremely flaggy	GR	gravelly
GRF	fine gravelly	GRC	coarse gravelly	GRV	very gravelly
GRX	extremely gravelly	SH	shaly	SHV	very shaly
SHX	extremely shaly	SY	slaty	SYV	very slaty
SYX	extremely slaty	CY	cindery	AY	ashy
SR	stratified	MK	mucky	PT	peaty
GY	gritty	GYV	very gritty	GYX	extremely gritty

Grade of Structure

1	weak	2	moderate	3	strong
4	very strong	5	weak and moderate	6	moderate and strong

Size of Structure

EF	extremely fine	VF	very fine	FF	very fine and fine
F	fine	FM	fine and medium	M	medium
MC	medium and coarse	CO	coarse		
CV	coarse and very coarse				

Structure Shape

PL	platy
LP	lenticular
PR	prismatic
COL	columnar
BK	blocky
ABK	angular blocky
SBK	subangular blocky
GR	granular
CDY	cloddy
CR	crumb
MA	massive
SGR	single grain
WEG	wedge

Dry Consistence

L	loose
S	soft
SH	slightly hard
SWH	somewhat hard
H	hard
VH	very hard
EH	extremely hard

Moist Consistence

L	loose
VFR	very friable
FR	friable
FI	firm
VFI	very firm
EFI	extremely firm

Other Consistence

WSM	weakly smeary
SM	smeary
MS	moderately smeary
D	deformable
SD	semideformable
B	brittle
R	rigid
VR	very rigid
CO	uncemented
VWC	very weakly cemented
WC	weakly cemented
SC	strongly cemented
I	indurated

Stickiness

SO	nonsticky
SS	slightly sticky
S	sticky
VS	very sticky

Plasticity

PO	nonplastic
SP	slightly plastic
P	plastic
VP	very plastic

Cementation Agent

H	humus
I	iron
L	lime
S	silica
X	lime and silica

Mottle Abundance Codes

F	few
C	common
M	many

Mottle Size Codes

1 fine
2 medium
3 coarse

Mottle Contrast Code

F faint
D distinct
P prominent

Surface Features

U coats
B black stains
D clay bridging
I iron stains
Q nonintersecting slickensides
L lime or carbonate coats
M manganese or iron-manganese stains
S skeletans (sand or silt)
X oxide coats
A skeletans over cutans
C chalcedony or opal
G gibbsite coats
K intersecting slickensides
P pressure faces
O organic coats
T clay films

Surface Feature Amount Codes

V very few
F few
C common
M many

Surface Feature Continuity Codes

P patchy
D discontinuous
C continuous

Surface Feature Distinctness Codes

F faint
D distinct
P prominent

Location of Surface Features

P on faces of peds
H on horizontal faces of peds
V on vertical faces of peds
Z on vertical and horizontal faces of peds
U on upper surfaces of peds or stones
L on lower surfaces of peds or stones
C on tops of columns
S on sand and gravel
R on rock fragments
F on faces of peds and in pores
N on nodules
M on bottoms of plates
B between sand grains
I in root channels and/or pores
T throughout

Boundary

A abrupt
C clear
G gradual
D diffuse
S smooth
W wavy
I irregular
B broken

Effervescence

1 slightly effervescent
2 stongly effervescent
3 violently effervescent
0 very slightly effervescent

Effervescence Agent Codes

H HCl (10%)
I HCl (unspecified)
P H_2O_2 (unspecified)
Q H_2O_2 (3 to 4%)

Field Measured Property Kind Codes

For organic materials

Column 1	Column 2	
F fiber	B unrubbed	R rubbed
H hemic	W woody	H herbacious
L limnic	S sphagnum	C coprogenous earth
S sapric	D diatomaceous earth	M marly
	F ferrihumic	U humilluvic
	O other	L sulfidic

For mineral materials

ON sand
OI silt
OA clay

pH

pM pH meter (1:1 H_2O)
pN pH (0.1 M $CaCl_2$)
pH Hellige-Truog
pL Lamotte-Morgan
pB Bromthymol blue
pC Cresol red
pP Phenol red
pT Thymol blue
pS soiltex
pY Ydrion

Soil Moisture Codes

D dry
M moist
V very moist
W wet

Quantity (Roots, Pores, Concretions)

VF very few
FF very few to few
F few
FC few to common
CM common to many
C common
M many

Size (Roots, Pores, Concretions)

M	micro	M1	micro and fine	V1	very fine
11	very fine and fine	1	fine	12	fine and medium
2	medium	23	medium and coarse	3	coarse
4	very coarse	5	extremely coarse	13	fine to coarse

Location of Roots

C	in cracks	M	in mat at top of horizon
P	between peds	S	matted around stones
T	throughout		

Shape of Pores

IR	interstitial	IE	filled with coarse material
IT	interstitial and tubular	IF	void between rock fragment
TU	tubular	TC	continuous tubular
TD	discontinuous tubular	TE	dendritic tubular
TS	constricted tubular	VS	vesicular
VT	vesicular and tubular	TP	total porosity

Kind of Concentrations

B1	barite crystals	B2	soft masses of barite
K2	soft masses of carbonate	K3	carbonate concretions
K4	carbonate nodules	C1	calcite crystals
C2	soft masses of lime	C3	lime concretions
C4	lime nodules	T2	worm casts
T3	insects casts	T4	worm nodules
A2	clay bodies	D1	mica flakes
D2	soft dark masses	D3	dark concretions
D4	dark nodules	E3	gibbsite concretions
E4	gibbsite nodules	G1	gypsum crystals
G2	masses of gypsum	F1	plinthite segregations
F2	soft masses of iron	F3	iron concretions
F4	ironstone nodules	M1	nonmagnetic shot
M2	soft masses of iron-manganese	M3	iron-manganese concretions
M4	magnetic shot	H1	halite crystals
H2	salt masses	S1	opal crystals
S2	soft masses of silica	S3	silica concretions
S4	durinodes		

Shape of Concentrations

C	cylindrical	d	dendritic	O	rounded	P	plate like
T	threads	Z	irregular				

Rock Fragment Kind Codes

Y mixed lithogoy
S sedimentary rocks
A sandstone
H shale
K organic fragments
O oxide-protected rock
I igneous rocks
B mixed sedimentary rocks
T siltstone
P pyroclastic rocks
F ironstone
M metamorphic rocks
L limestone
E ejecta
R saprolite

Rock Fragment Size Codes

1 pebbles
2 cobbles
3 stones
4 boulders
5 channers
6 flagstones
C 20- to 75-mm fragments

APPENDIX B

LABORATORY 3 AMMONIUM TEST

This appendix is an excerpt of a letter from the preparation laboratory manager of Laboratory 3. A test was made on soil samples to determine whether or not an ammonia leak in the cold storage facility contaminated any of the samples from the Northeastern soil survey.

December 18, 1985

QA Manager
Lockheed-EMSCO
1050 E. Flamingo Road
Suite 120
Las Vegas, Nevada 89109

This letter is to confirm for your records the results of the ammonium test performed on soil samples, as discussed in our telephone conversation.

At our initial observation visit I pointed out a leak of ammonia gas coming from a room adjacent to the cooler in which we would be storing NADSS samples. In order to determine the extent of possible contamination of the NADSS samples by ammonia, the following procedure was followed.

On September 11, 1985, 3 sets of the same soil samples were placed in the storage cooler. Each set was repeated in triplicate and represented the following:

1. one set of uncovered soils
2. one set of soils enclosed in plastic liners only
3. one set of soils enclosed in plastic liners and canvas bags

Subsamples of these soils had been previously tested for ammonium content by the Nessler Reagent Colorimetric test (2M KCl extraction), the standard ammonium test used by the laboratory (Greweling and Peech, 1960, Chemical soil tests, Cornell Exp. Sta. Bull. 960). These preliminary results showed that control samples were below the ammonium detection limit at the beginning of the experiment.

On October 9, 1985, each set of soils placed in the cooler was removed and analyzed for ammonium by the Nessler Reagent test. Results showed the following:

1. unccovered soils had 10 ppm ammonium
2. soils in plastic liners only were below the ammonium detection limit
3. soils in plastic liners and canvas bags were below the ammonium detection limit.

According to these results, it appears that no change occurred in the soils covered with plastic, and canvas and plastic from the original amount of ammonium. When left uncovered in the cooler, 10 ppm ammonium was adsorbed by the soil, assumably due to the ammonia leak in the adjacent room.

Soil samples prepared by the department of the NADSS project were placed in 2 plastic liners, 1 canvas bag, an additional plastic liner, and a cardboard box before being placed in the cooler for storage. Thus it can be stated that ammonium contamination of these soils would be nil or negligible.